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The American Mineralogist

Journal of the Mineralogical Society of America

Vol. 36 MAY-JUNE, 1951 Nos. 5 and 6

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Volume 5, Part 3

Sponsored by **The Walker Mineralogical Club**

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Published bi-monthly by the Society

Mineralogical Society of America

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The enlarged issues of this journal for 1951 are made possible by a grant from the Penrose Fund of the Geological Society of America.

The American Mineralogist—Journal of the Mineralogical Society of America

A journal containing articles on mineralogy, crystallography, petrography, and allied sciences, issued every two months. Contributions are invited from everyone.
Office of Publication, Mineralogical Laboratory, Ann Arbor, Mich.

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Addl. C's	1.20	2.20	3.20	3.60	4.40	4.80	5.60	6.00	2.40

Cover Composition $1.55.

Sent to all members and fellows of the Mineralogical Society of America. Subscription price, $4.00 per year (single copies of normal issues, $1.00 plus postage).

Entered as second class matter at the post office at Menasha, Wis., under Act of March 3, 1879. Acceptance for mailing at the special rate of postage provided for in section 1103, Act of Oct. 3, 1917, paragraph 4 section 429 P. L. & R. authorized March 13, 1922.

Notices of change of address, orders, and remittances should be sent to Dr. Earl Ingerson, U.S. Geological Survey, Washington 25, D. C.

Printed by the George Banta Publishing Company, Menasha, Wisconsin

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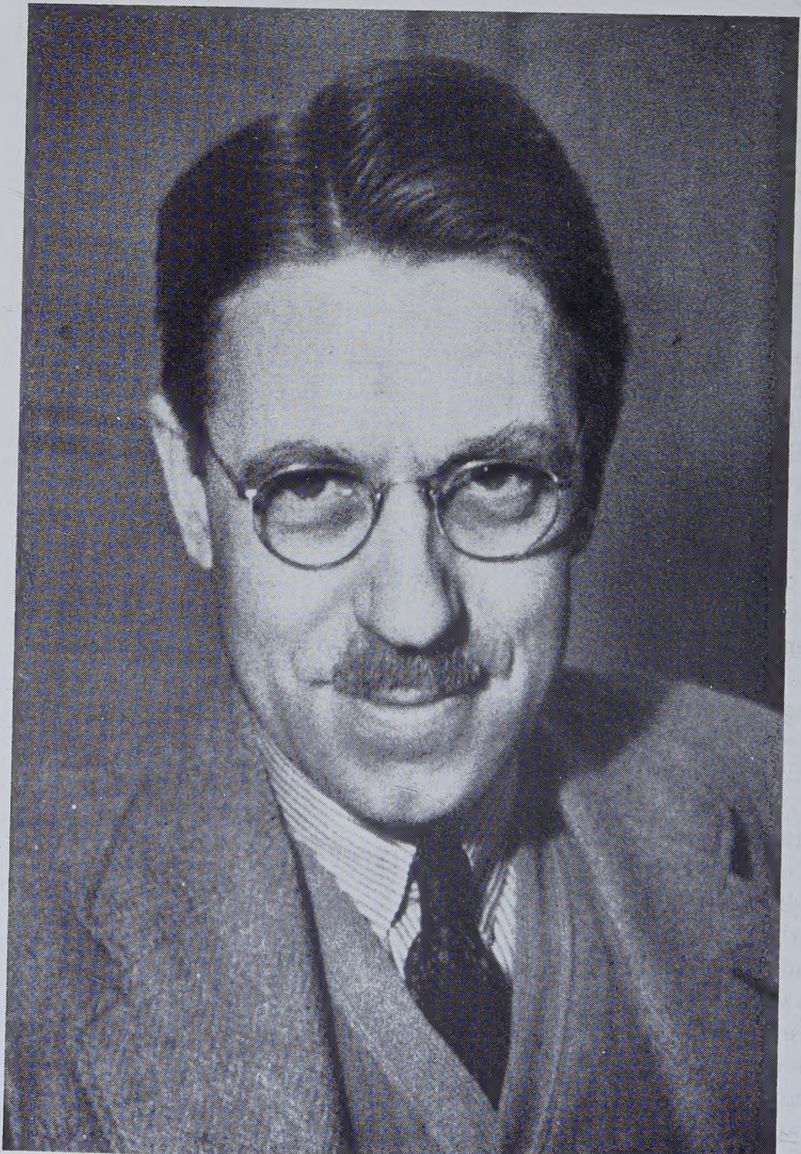

Martin Alfred Peacock
1898–1950

THE AMERICAN MINERALOGIST

JOURNAL OF THE MINERALOGICAL SOCIETY OF AMERICA

Vol. 36 MAY-JUNE, 1951 Nos. 5 and 6

MEMORIAL OF MARTIN ALFRED PEACOCK*

CHARLES PALACHE, *Cambridge, Massachusetts*

The death of Martin Peacock on October 30, 1950 has left a gap in the ranks of American mineralogists that cannot soon be filled. The science has lost a successful teacher and a leader in research at the height of his powers. His colleagues all across the land mourn a dear and faithful friend.

His illness ran its course within a year. After a first operation late in 1949 Peacock knew that he had not long to live. With characteristic calm he faced this unkind fate without repining; compelled to give up teaching he turned to the projects he had in hand and completed many of them or placed them in the way of completion by others. In May when he drove from Toronto to Cambridge with two of his students he seemed to the writer, in whose home he visited, to be in excellent spirits and unexpected vigor. But he took cold on the return trip so that his reserve of strength was lowered. A trip to Southern California by air later in the summer was apparently accomplished without undue fatigue. The final illness was mercifully brief.

Peacock was born in Edinburgh, Scotland, January 15, 1898. His father, Alfred Norman Peacock, was an engineer from whom he doubtless inherited some of the mechanical skill with tools and the keen sense of order in all his work and surroundings that was so characteristic. His early home was in Edinburgh but later the family moved to Glasgow and there he attended High School and entered the University of Glasgow in 1915. His work was interrupted by the incidence of the first World War; he enlisted in January 1917 and was commissioned 2nd Lieutenant in the Royal Flying Corps in May. He went to the front in September but his plane was shot down in October and he was in a German war prison camp for more than a year until the end of the war. Demobilized in 1919, he returned to Glasgow and finished his course with the B.Sc. degree in 1922. He also studied music in Glasgow and took his Licentiate in pianoforte at the Royal Academy of Music in

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* Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 326.

London in 1923. Although he taught it only a short time, his music was a principal recreation all his life.

After graduation Peacock took up the study of petrography at Glasgow under Professor J. W. Gregory. He received a Carnegie Research Fellowship by the aid of which, in addition to teaching, he carried on for four years a study of the igneous rocks of Iceland. This began with the collections of Iceland rocks made a century before by the Scotch geologist Sir C. S. Mackenzie and preserved in the Glasgow Museum. There followed a summer of field work in Iceland with G. W. Tyrrell and a brief visit to the Faeroe Islands. His first publications give the results of these studies and in 1925 he received the degree of Ph.D. based on one of them as a thesis. His third degree from Glasgow was the D.Sc., received in 1932.

In 1926 Peacock was named a Commonwealth Fund Fellow at Harvard University and this appointment was twice renewed. At Cambridge he continued petrographic study under Professor Larsen; developed an interest in geomorphology which had begun when he travelled amid the fiords of Iceland and the Faeroes; and began the study of crystallography with the writer. He spent the summer of 1927 with H. A. Powers in the Modoc lava field of Northern California and wrote the general physiographic description of the region.

Peacock's interest in the subject that became his life work, crystallography, began early in his Harvard residence. He quickly learned the use of the Goldschmidt goniometer, rejoicing in the graphical presentation of the measurements which his exquisite draughtsmanship made doubly effective. So intent and expert did he become in this work that before the first year was over I had turned over to him the intricate problem of the crystal form of the mineral calaverite which I had for years tried in vain to solve. His exact plots of the measurements of many crystals led to a partial solution of the involved relations. Feeling myself unable to interpret or clarify his results, I sponsored through the Department funds a sojourn to Heidelberg in the summer of 1929 for consultation with Victor Goldschmidt, who had also been working fruitlessly on calaverite. Peacock became at once a favorite of both Goldschmidt and his wife; his skill with the pencil and at the piano were alike keys to their confidence and love. The resulting joint paper on calaverite, while not solving the riddle, was yet the clearest presentation yet made of the observations on this most unusual mineral.

Returning to America in 1929, Peacock, his Commonwealth Fund Fellowship at an end, accepted a temporary position at the University of British Columbia at Vancouver. He taught geology and geography, first as Lecturer, then as Assistant Professor. He took advantage of his residence on the Canadian west coast to continue the study of fiords begun in the North Atlantic and his resulting paper, based on field trips along the coast in the summers of 1930, 1931, and 1932, was an important contribution to the subject. Its publication in 1935 in the *Bulletin of the Geological Society of America* brought to the fore his interest in the literary form of scientific publications. I find in the Department files a copy of a letter written by Peacock in 1935 to the Board of Editors of the Geological Society of America regarding *Style in References*. This was mimeographed and circulated to the Board and served as a basis for discussion. I believe it was the primary influence which led to the adoption of the present form of reference used in the publications of the society. His interest in editorial matters was not by any means ended with this first venture; he was a stickler for correct form in every paper that issued from our department while he was with us and at the end of that period was in a position to make particular use of his talent.

His appointment in Vancouver coming to an abrupt and unforeseen end in 1932 owing to the hard times of the early thirties, Peacock found himself without a job and in difficult circumstances as he could not return to the United States where openings were most likely without having a definite appointment. This stalemate was broken by an appointment as Research Fellow at Yale University. After he had been there a few months I was able to offer him a more permanent position as one of the Research Assistants collecting and organizing material for the proposed revision of Dana's *System of Mineralogy*. He threw himself into this work with enthusiasm, organizing the data into new and improved forms of presentation; restating the crystallography of many mineral species; and publishing in rapid succession a series of mineralogical papers as may be seen by consulting the bibliography for the years from 1933 to 1938.

In the summer of 1933 an urgent call upon me for help by Frau Goldschmidt to enable her ailing husband to complete pending work led the Department of Mineralogy to finance for Peacock a residence of three months in Heidelberg. Although the beloved master had died before Peacock reached Heidelberg, it was possible for him to edit and complete several papers which were under way and to bring much aid and comfort to the widow.

Peacock was at Harvard from 1932 to 1937. Work on the actual preparation of manuscript for the Dana revision began in 1936 when the grant from the Geological Society for that purpose became available. Peacock and Berman were active joint editors under the advisory direction of Professor Ford of Yale and myself. It is to Peacock's editorial skill that the seventh edition of Dana owes much of its form, although his name does not appear on the title page as editor. After working with Berman for a year or more he was called to the chair of Mineralogy at Toronto University in 1937. He kept up active participation in the Dana work for some time but that became impracticable as his work at Toronto developed and later he and his students helped as they could; but he prepared no final manuscript.

During Peacock's residence at Harvard, x-ray equipment for mineral study began to be installed. With his skill at all mechanical instruments he soon became familiar with this new technique, and a visit of two weeks to the Geophysical Laboratory in Washington in 1935 served to round out his knowledge. When he went to Toronto he designed and installed a very complete laboratory for x-ray studies, which was in constant use by his students and himself. How he acquired the familiarity with the theory of crystal structures and their instrumental determination, which he soon evidenced, I never knew. He seemed to absorb it without instruction; but it became of the most fundamental importance to him in crystal study and was basic in his teaching of crystallography.

At Toronto, Peacock added to his teaching of elementary mineralogy the direction of research by a group of graduate students whose work was often published jointly with him. These men became loyal friends, and their thorough training is evidenced by the positions of responsibility as teachers or research workers many of them now hold. As editor of the *University of Toronto Studies, Geological Series*, from 1944 he continued the series of *Contributions to Canadian Mineralogy*. His last work during the early part of the summer of 1950 was to see through the press, with all his usual careful supervision, the latest volume of this series which constitutes Nos. 5 and 6 of Volume 35 of *The American Mineralogist.*

It was a great satisfaction to Dr. Peacock that, when he was promoted in 1946 to a full Professorship at Toronto, his title was Professor of Crystallography and Mineralogy. The writer is not aware of any other appointment in America so emphasizing the importance crystallography has come to have not only in mineralogy but in a host of related fields of science.

Peacock established four new mineral species: parawollastonite, pararammelsbergite, hedleyite, and montbrayite. He redefined fundamentally the minerals maucherite, parkerite, heazlewoodite and hauchecornite. His name will, I believe, remain longest in the memory of mineralogists for the rigor of his descriptions, the conciseness of his style of presentation, and for the emphasis which he placed upon the structural elements of the crystal in the description of a mineral species.

In 1937 Dr. Peacock was married to Katharine Louisa West of Glens Falls, New York. She and two daughters survive him.

Peacock was a fellow of the Royal Society of Canada; a fellow of the Mineralogical Society of America and its President in 1948; a fellow of the Geological Society of America and vice president in 1949; a member of the American Geographical Society, of the American Crystallographic Association, of the Mineralogical Society of Great Britain, of the Société Français de Mineralogie, and of the Walker Mineralogical Club of the University of Toronto, in the affairs of which he took an active part.

I cannot conclude this rather personal sketch of the life of Martin Peacock without an expression of my own sense of loss in his untimely passing. His gentleness and kindly affection were those of a son. To have him come into the home and sit down at the piano to play one of his favorite Beethoven sonatas gave me unmatched happiness. I spent many congenial hours with him at the workbench, putting into shape simple instruments or pieces of furniture which his pencil had sketched in the minutest detail. The plane he had sharpened was the truest and keenest; his chisel was unerring in fitting tenon to mortice. It was a joy to see him work.

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1924

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A contribution to the petrography of Iceland: *Trans. Geol. Soc. Glasgow,* **17,** 273–333.

1926

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1928

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1929

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1930

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1931

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1932

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1933

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1935

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Topaz from Devil's Head, Colorado: *Am. Mineral.* **20,** 354–363.

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1936

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1937

On rosenbuschite: *Norsk Geologisk Tidsskrift*, **17,** 17–30.

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1938

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1939

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1940

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1941

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1942

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(with E. W. Nuffield) Recrossing axial plane dispersion in goethite: *Univ. Toronto Studies, Geol. Ser.*, **47,** 53–61.

Studies of mineral sulpho-salts—VI—aikenite: *Univ. Toronto Studies, Geol. Ser.*, **47,** 63–69.

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1943

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1944

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1945

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(with H. V. Warren) Hedleyite, a new bismuth telluride from British Columbia, with notes on wehrlite and some bismuth-tellurium alloys: *Univ. Toronto Studies, Geol. Ser.*, **49,** 55–69.

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1946

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1947

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1948

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1949

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The teaching of morphological crystallography: *Am. Mineral.*, **34,** 291–292.

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1950

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1951

(with R. M. Thompson, J. F. Rowland and L. G. Berry) Empressite and "stuetzite": *Am. Mineral.* (in press).

STUDIES OF RADIOACTIVE COMPOUNDS: I—VANDENBRANDEITE[1]

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University of Toronto, Toronto, Canada

ABSTRACT

Vandenbrandeite is triclinic with a 7.84, b 5.43, c 6.09 kX; α 91°52′, β 102°00′, γ 89°37′ and cell content $2[CuUO_4 \cdot 2H_2O]$. An angle table has been calculated and powder data are given.

The usual method of defining crystal systems in terms of crystallographic axes is inadequate; the classification of crystals must rest on the symmetry that has its origin in the atomic arrangements. A review of rules for orienting triclinic crystals suggests that the one important standardization is in the selection of the conventional structural cell, which has as edges the three shortest noncoplanar translations in the lattice. This cell is easily recognized from its dimensions and angles regardless of the setting, and is readily reoriented for a special purpose. The preferred orientation of the cell should if possible, have α and β obtuse, and $a<b$. It should be designed to best describe the mineral. Any noteworthy property, such as structural or morphological analogy to other minerals should influence the choice of setting; in the absence of an outstanding feature morphological crystallographers will probably continue to designate some prominent direction within the crystal as the c axis.

Vandenbrandeite is a hydrous copper uranate which has been found only in the Katanga district of the Belgian Congo. The mineral was first described and named by Schoep (1932), who studied material from Kalongwe. Thoreau (1933) later reported it from Shinkolobwe under the name uranolepidite. The published observations of these two men leave little doubt of the identity of vandenbrandeite and uranolepidite. We are indebted to them for two chemical analyses and a description of the physical and optical properties that is adequate for future identification of the mineral. However, the crystallography of vandenbrandeite has not been fully studied. Crystal measurements leading to axial ratios have not been made and the triclinic symmetry has been inferred on the basis of optical examinations. Hence the mineral offered an interesting and useful study.

Through the kindness of Professor V. B. Meen, we were fortunate to obtain the loan of a specimen (ROM M18598) from the type locality of Kalongwe which had been presented to the Royal Ontario Museum of Geology and Mineralogy by M. Thoreau. This specimen is composed of massive dark green vandenbrandeite containing minute (to $\frac{1}{3}$ mm.)

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[1] Extracted from a Ph.D. thesis.

[2] Research Council of Ontario scholar.

[3] Assistant Professor, Department of Geological Sciences.

tabular crystals of the mineral in vugs. The crystals are partially covered by a light brown deposit which was identified as kasolite with an x-ray powder photograph.

The optical properties proved most useful in establishing the identity of our material with that studied by Schoep and Thoreau. Under the polarizing microscope fragments of the mineral show moderate pleochroism with colours varying from yellow-green to blue-green. Fragments lying on the excellent cleavage are blue-green in colour, not pleochroic and have a very low birefringence. They are within 5° of being perpendicular to an optic axis and it is therefore possible to make a fairly accurate determination of the Y index. The optical data we obtained are compared below with those of Schoep and Thoreau:

	Schoep	Thoreau	Milne and Nuffield
X	1.77 ± 0.02	1.76	1.765 ± 0.005
Y	1.78 ± 0.02	—	1.792 ± 0.002
Z	1.80 ± 0.02	1.80	—
$2V$	—	large	near 90°

Thoreau gave the optical sign as negative but Schoep's indices of refraction require a positive sign. Optical measurements on our material were made difficult by a great number of inclusions. Schoep considered these to be kasolite and this is supported by the presence of this mineral on the crystal faces of vandenbrandeite. In addition to solid inclusions, many rounded liquid inclusions containing gas bubbles were seen.

The cleavage referred to above is parallel to (110) in our setting and is the only one we observed. The optical study indicates that this is the (001) cleavage of Schoep, who recorded a second direction and suggested the possibility of a third. Thoreau also observed three cleavages which he said caused the mineral to break into laths. It is possible that Schoep and Thoreau had better material than we did but we believe that the presence of the two additional cleavages should be accepted with reservations.

A study of the crystals showed that the tabular faces are characteristically rounded and poorly reflecting. However, some crystals have a zone of several well developed faces, the axis of which is inclined at about 13° to the normal of the tabular face. On the chosen crystal this axis, which was later designated as $c[001]$ was utilized as the rotation axis to obtain two-circle goniometric measurements of the faces, as well as rotation, and zero and first layer Weissenberg x-ray photographs. Consideration of the gnomonic and reciprocal lattice projections revealed triclinic symmetry and indicated that the rotation axis is one of the three shortest lattice translations. It was now possible to select the remaining two translations in the lattice and to locate their direction in the crystal. To assure more accurate cell constants zero layer Weissenberg photographs were prepared by turning the crystal about these two directions. Measurement of the films gave $d(100)$ 7.67, $d(010)$ 5.43, $d(001)$ 5.95kX;[1] $\alpha^*88°10'$, $\beta^*78°00'$, $\gamma^*90°00'$. From these values the reciprocal and direct cell elements were calculated:

Reciprocal cell		Direct cell	
$a^*=0.1304$	$\alpha^*=88°10'$	$a=7.84$ kX	$\alpha=91°52'$
$b^*=0.1842$	$\beta^*=78\ 00$	$b=5.43$	$\beta=102\ 00$
$c^*=0.1681$	$\gamma^*=90\ 00$	$c=6.09$	$\gamma=89\ 37$

The morphology of the crystal gives no reason to suspect the absence of a symmetry center. Therefore the mineral has been placed in spacegroup $P\bar{1}$. The usual x-ray powder data obtained on a clean sample is presented in Table 1, and the photograph itself is reproduced in Fig. 1.

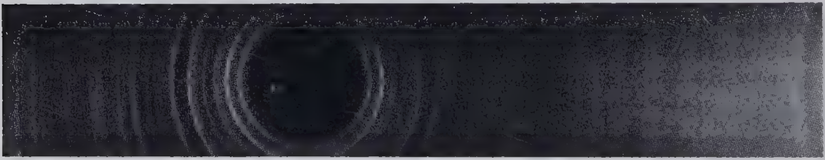

FIG. 1. Vandenbrandeite: X-ray powder print; Cu/Ni; 1 mm.$=1°\vartheta$.

TABLE 1. VANDENBRANDEITE: X-RAY POWDER DATA

Triclinic-$P\bar{1}$; a 7.84, b 5.43, c 6.09; α 91°52′, β 102°00′, γ 89°37′

I	d(meas.)	I	d(meas.)	I	d(meas.)	I	d(meas.)
4	5.05 kX	2	1.963	½	1.417	½	1.066
10	4.28	1	1.909	½	1.395	½	1.053
2	3.84	3	1.847	2	1.345	½	1.042
2	3.71	½	1.819	½	1.330	½	1.035
2	3.32	1	1.789	3	1.314	½	1.026
1	3.06	½	1.747	½	1.297	½	1.018
8	2.91	1	1.705	½	1.273	½	1.010
½	2.76	½	1.682	2	1.250	½	1.001
4	2.55	½	1.654	½	1.243	1	0.988
½	2.47	2	1.621	1	1.224	½	0.976
½	2.39	½	1.576	½	1.195	½	0.966
1	2.35	1	1.554	½	1.171	1	0.952
1	2.28	1	1.524	½	1.154	1	0.942
½	2.19	1	1.495	½	1.127	1	0.937
½	2.15	3	1.469	2	1.094	1	0.929
3	2.09	½	1.435	2	1.074	½	0.925

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[1] Using Cu$K\alpha=1.5374$ kX.

The choice of the setting of vandenbrandeite has been influenced by the close approach to monoclinic lattice geometry and by the tabular habit. The axis which is very nearly perpendicular to the other two was chosen as the *b* axis. Next the *a* axis was selected from the remaining two possibilities in order to describe the broad face as (001). Finally the positive directions of the axes were so chosen as to make α and β obtuse.

In this setting the crystals are tabular parallel to *c*(001) and somewhat elongated parallel to the edge $c(001)\wedge m(110)$. The habit is portrayed in Fig. 2. The relative dimensions of the faces have been preserved as far as possible in the drawing, but the crystal has been idealized by the addition of the assumed center of symmetry. The crystals are commonly attached by *m*(110) and grow outward in a direction defined by the edge $c(001)\wedge M(1\bar{1}0)$. Examination of the crystals gives one the impression that the mineral would exhibit a lozenge-shaped habit controlled by *m*(110) and $M(1\bar{1}0)$ under conditions permitting greater freedom of crystal growth thus heightening the illusion of monoclinic symmetry.

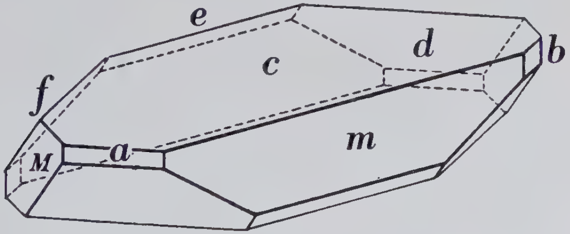

FIG. 2. Vandenbrandeite: crystal from Kalongwe, Katanga.

Crystals are so implanted on the massive material as to show *m*(110), which is the excellent cleavage, to advantage. This probably influenced Schoep (1932) to describe it as *c*(001). The crystal sketch which accompanies his description shows four other forms to three of which he assigned indices without deriving axial ratios from crystal measurements. It is possible to identify all the forms as follows:

Schoep	Structural
(001)	*m*(110)
$(1\bar{1}0)$	*c*(001)
(100)	*a*(100)
(110)	$d(10\bar{1})$
not named	$M(1\bar{1}0)$

The crystal faces are far from perfect and as a result, measurements on the two-circle goniometer proved inconsistent. On the other hand the rotation and zero-layer Weissenberg photographs about the three axial directions were sharp and yielded good results. Consequently the cell constants were used to calculate axial ratios and reciprocal and projection elements and finally the angle table for the observed forms (Table 2). As a check on our calculations Table 3 has been prepared giving a comparison between the measured and calculated two-circle angles, ϕ and ρ for all the faces on the crystal. It is to be noted that the agreement between corresponding angles is usually within one degree and that there is no doubt that correct Miller indices have been assigned to the faces.

TABLE 2. VANDENBRANDEITE: $CuUO_4 \cdot 2H_2O$

Triclinic, $P\bar{1}$

$a:b:c = 1.443:1:1.120;\ \alpha = 91°52', \beta = 102°00', \gamma = 89°37'$
$p_0:q_0:r_0 = 0.776:1.096:1;\ \alpha^* = 88°10', \beta^* = 78°00', \gamma^* = 90°00'$
$p_0' = 0.794,\ q_0' = 1.121,\ x_0' = 0.2127,\ y_0' = 0.0327$

Form	ϕ	ρ	A	B	C
$c(001)$	81°15′	12°09′	78°00′	88°10′	0°00′
$b(010)$	180 00	90 00	90 00	180 00	88 10
$a(100)$	90 00	90 00	180 00	90 00	78 00
$m(110)$	35 18	90 00	54 42	35 18	81 35
$M(1\bar{1}0)$	144 42	90 00	54 42	144 42	84 36
$f(0\bar{1}1)$	168 57	47 57	81 49	136 47	48 38
$d(\bar{1}01)$	−86 47	30 11	120 08	88 23	42 08
$e(\bar{1}\bar{1}2)$	−160 46	29 12	99 15	117 26	36 22

TABLE 3. VANDENBRANDEITE

Face	Measured		Calculated	
	ϕ	ρ	ϕ	ρ
$c(001)$	82°29′	12°25′	81°15′	12°09′
$\bar{b}(0\bar{1}0)$	180 00	90 00	180 00	90 00
$a(100)$	−87 51	90 00	−90 00	90 00
$m(110)$	35 20	89 43	35 18	90 00
$\bar{m}(\bar{1}\bar{1}0)$	−145 39	90 00	−144 42	90 00
$M(1\bar{1}0)$	144 36	90 00	144 42	90 00
$\bar{M}(\bar{1}10)$	−35 35	90 00	−35 18	90 00
$f(0\bar{1}1)$	168 54	46 55	168 57	47 57
$d(\bar{1}01)$	−87 56	29 12	−86 47	30 11
$e(\bar{1}\bar{1}2)$	−161 42	30 00	−160 46	29 12

Vandenbrandeite and the Bravais Rule

The "Law" of Bravais states that the importance (size and frequency) of crystal forms is proportional to reticular densities, or the spacings, of the corresponding lattice planes; this implies that the rate of growth is proportional to lattice row density. Donnay and Harker (1937) extended this rule to include the effects of space group symmetry operations involving translation on the lattice spacings. The rules coincide for triclinic space groups since screw axes and glide planes are not involved. Mineral examples (Nuffield & Peacock, 1945) have been presented which support with certain exceptions, the Bravais rule in its modified form. Vandenbrandeite is the case of a mineral that differs remarkably from the ideal Bravais crystal.

To test the observed importance of crystal forms on vandenbrandeite against the theoretical importance demanded by the Bravais rule, Table 4 has been prepared. This table gives all the calculated spacings in decreasing order to include the observed forms. The Bravais rule predicts that the three most important forms shall be (100), (001) and (010), decreasing in that order. The habit should be tabular parallel (100) with elongation if any parallel to *b*[010]. Actually (100) and (010) are among the smallest faces; crystals are tabular parallel to (001) and elongated parallel to the edge (001)$\wedge$(110) which is not an edge of the conventional cell. The appearance of the form ($\bar{1}\bar{1}2$) is another anomaly. It seems that the only point in support of the Bravais rule is the fact that 7 of the 8 observed forms are among the 8 largest spacings.

TABLE 4. VANDENBRANDEITE: CALCULATED SPACINGS

α^* 88°10′, β^* 78 00, γ^* 90 00
a 7.84, *b* 5.43, *c* 6.09kX; α 91°52′, β 102 00, γ 89 37

Plane	d_{kX}	*Form*	*Plane*	d_{kX}	*Form*
(100)	7.67	*a* —small	($\bar{1}$11)	3.74	—
(001)	5.95	*c* —broad, dominant	($\bar{2}$01)	3.58	—
(010)	5.43	*b* —very small	(1$\bar{1}$1)	3.39	—
(10$\bar{1}$)	5.31	*d* —large	(111)	3.32	—
(1$\bar{1}$0)	4.43	*M*—large, smaller than *m*	(210)	3.13	—
(110)	4.43	*m* —second largest face	($\bar{2}$11)	3.02	—
(101)	4.26	—	($\bar{1}$02)	2.99	—
(01$\bar{1}$)	4.08	*f* —medium	(201)	2.96	—
(011)	3.95	—	(102)	2.69	—
($\bar{1}\bar{1}$1)	3.85	—	($\bar{1}\bar{1}$2)	2.66	*e*—small

The lozenge-shaped crystal outline of vandenbrandeite is reminiscent of the habit displayed by certain base-centred monoclinic minerals like plagionite and semseyite (Nuffield & Peacock, 1945). The crystal outlines of these two minerals are due to the predominant development of the (*hhl*) and ($\bar{h}hl$) zones and are in accord with the modified rule of

Bravais. The extinction condition characteristic of base-centring, (*hkl*) present only with $h+k$ even, halves the effective lattice spacings of (100) and (010) and consequently increases the relative theoretical importance of (*hhl*) and ($\bar{h}hl$) forms. Vandenbrandeite is analogous in that the crystallographic elements are pseudo-monoclinic; furthermore, two forms in the (*hhl*) and ($h\bar{h}l$) zones, *m*(110) and *M*(1$\bar{1}$0) dominate and largely determine the crystal outline. However, the Weissenberg photographs do not indicate a centred conditioned in the lattice. Consequently we are left with the interesting speculation that the structure falls just short of a base-centred condition, but nevertheless may influence crystal growth as though this condition were realized. However, many anomalies remain, in particular the absence of the 4 unit (*hkl*) pinakoids.

Despite the case just cited, a sufficient number of mineral examples are to be found in the literature to indicate that form development is often in accordance with the Bravais rule; many exceptions to this rule can be explained by the Donnay-Harker modification. It would however, be too much to expect crystal growth to proceed in all or even in most cases according to a rule based on purely geometrical considerations and which " . . . does not involve any consideration of the special positions that the atoms may occupy, nor does it take into account their nature, their charge, their bonds, or any other physical concept" (Donnay & Harker, 1937, p. 455). Buerger (1947) has pointed out that the characteristics of the particles which arrive at the crystal surfaces during growth are a fundamental factor in determining crystal habit. It would seem therefore, that these rules are but first approximations in the attempt to evaluate the influence of the atomic structure upon its form development. Instances of good agreement between crystal habit and the Bravais-Donnay-Harker rule suggest that in these cases the effects of the packing and bonding of atoms and of the environment of the crystal may be equated to the lattice in terms of the modified rule of Bravais. It appears that a study of the atomic structures of non-conforming crystals is necessary to broaden the scope of the rules.

Composition and Cell Content

Vandenbrandeite has been analyzed by Schoep (1932), and Boubnoff (in Thoreau, 1933). Both analyses (Table 5) indicate the presence of impurities and this is confirmed by the observation during this study of kasolite coating the crystal faces and of numerous inclusions within the crystals. Schoep adjusted his analysis by deducting PbO as kasolite ($3PbO \cdot 3UO_3 \cdot 3SiO_2 \cdot 4H_2O$) and by regarding Fe_2O_3 and P_2O_5 as impurities. In this paper Schoep's analysis has been adjusted by the withdrawal of PbO as kasolite on the basis of the now generally accepted formula for this mineral ($PbO \cdot UO_3 \cdot SiO_2 \cdot H_2O$). Each analysis with its accompanying measured specific gravity has been used to calculate the cell contents. The results are given in Table 5. Clearly CuO and UO_3 approach 2. The values are below 2 probably because the measured specific gravities are too low, a logical deduction in view of the numerous liquid inclusions. The water content in both analyses is between 4 and 5 It is likely that the ideal value is 4 and the measured value is higher because of the abundance of liquid inclusions. The results point to the ideal cell content $2[CuO \cdot UO_3 \cdot 2H_2O]$ and this is the commonly accepted composition (Dana, 1944, p. 632). The calculated specific gravity is 5.26.

TABLE 5. VANDENBRANDEITE: ANALYSIS AND CELL CONTENTS
CELL VOLUME 253.5kX^3

	I			II			III	
	A	B	C	A	B	C	A	B
CuO	15.78	18.77	1.87	18.98	19.20	1.91	19.81	2
UO_3	65.45	70.69	1.78	70.40	71.23	1.85	71.22	2
H_2O	9.25	10.54	4.41	9.46	9.55	4.08	8.97	4
PbO	4.69	—	—	—	—	—	—	—
SiO_2	1.66	—	—	0.28	—	—	—	—
Total	98.59[1]	100.00		99.95[2]	100.0		100.00	
G	4.96 (meas.)			5.03 (meas.)			5.26 (calc.)	

I. Kalongwe, Katanga district, Belgian Congo. A. Anal. Schoep (1932); [1]incl. Fe_2O. 1.55, P_2O_5 0.21. B. Analysis corrected by deducting kasolite ($PbO \cdot UO_3 \cdot SiO_2 \cdot H_2O$), SiO_2, Fe_2O_3 and P_2O_5 and summing to 100 per cent. C. Cell contents calculated for the cell volume and measured specific gravity 4.96.

II. Shinkolobwe, Katanga district, Belgian Congo. A. Anal. Boubnoff (in Thoreau, 1933); [2]incl. CaO 0.26, MgO 0.57. B. Analysis recalculated to 100 per cent after deducting SiO_2, CaO and MgO. C. Cell contents calculated for the cell volume and measured specific gravity 5.03.

III. A. Calculated weight per cent and specific gravity for $2[CuUO_4 \cdot 2H_2O]$. B. Ideal cell contents.

THE CONVENTIONAL TRICLINIC CELL AND ITS SETTING

During the course of this study we had occasion to examine some of the literature concerned with the definition and description of a triclinic species. It became apparent that a summary and discussion of recent published works on the subject would be of value at the present time.

The Triclinic System

Crystal systems are usually defined in terms of the geometry of unit cells, or with reference to crystallographic axes by authors of textbooks.

The following abstracts illustrate this common practice. "Crystals lacking symmetry of any kind naturally have the most 'general' type of unit cell, the three axes of which are all inclined to each other at different angles and unequal in length. The addition of a centre of symmetry does not alter the situation . . . " (Bunn, 1945, p. 47). "In the triclinic system there are three crystallographic axes of unequal length that make oblique angles with each other" (Hurlbut, ed. 15, p. 57). "The triclinic system includes all crystals which are correctly referred to three unequal crystal axes intersecting each other at unequal angles. In general all three angles are oblique, but by chance one of them may be a right angle" (Winchell, 1942, pp. 106–107). "In the Triclinic System the crystallographic axes are all unequal and none is at right angles to another" (Rutley, 1936, p. 114). "The three axes in this system (Anorthic) being not only unequal in length, but also inclined to each other at different angles, none of which is a right angle . . . " (Miers, 1929, p. 87). It is not unusual to find that the crystal systems are distinguished on the basis of the geometry of space lattices. These expressions represent a peculiar anomaly in view of the fact that the systems are usually regarded as groups of crystal classes which in turn are almost universally regarded as symmetry combinations. Evidence will be presented in the following paragraphs to show that descriptions of systems based on the crystallographic axes are inadequate.

A crystal may be considered as a homogeneous structure consisting of an infinite number of identical units (unit cells) stacked in three dimensions; this implies that the structure may be attained by repeated and equal translations of the unit along its three edges.

Identical arbitrary points in the structure form a regular three-dimensional framework in space known as a point space lattice. The geometry of the crystal is in harmony with the geometry of this lattice. It follows that the crystallographic axes of the properly chosen morphological unit coincide in direction with the edges of the structural unit and with certain translation directions within the lattice. It can be readily appreciated that the crystallographic axes, the lattice and the cell edges are devices that define merely the geometry of the unit cell since they do not take full account of the symmetry of the atomic structure; therefore the symmetry of these geometrical devices need not be the same as the symmetry of the structure. The positions relative to one another of the identity points within the structure define the shape and therefore the symmetry of the lattice. This symmetry cannot be lower than that of the structure; in fact its symmetry is never lower than that of the highest symmetry class in the system of crystallization (or subsystem if hexagonal). Actually the identity points may outline a lattice that has higher symmetry than the structure; it is conceivable for a triclinic crystal to have a lattice with cubic geometry and therefore cubic-like crystals referable to three equal axes inclined to each other at right angles. It becomes apparent that crystal systems cannot be accurately defined in terms of either the symmetry or the geometry of lattices, crystallographic axes or unit cells. The classification of crystals into systems and classes must rest on the symmetry of the atomic arrangements.

It is evident then that the above abstracted statements refer to specific cases. The general and therefore the more complete statement of a crystal system should give a description of its characteristic symmetry and the limitation this symmetry imposes on the generality of the geometry of unit cells, lattices or crystal axes. For example, triclinic minerals have only one-fold symmetry axes ($\bar{1}$, 1); this symmetry imposes no restriction on the lengths of the 3 axes of the unit cell or their angular inclinations to one another.

The Conventional Triclinic Cell

There is general agreement among crystallographers that except in special cases, the properly chosen triclinic structural cell has as edges the three shortest noncoplanar translations in the lattice. In the geometrical sense there are two types of triclinic cells with oblique angles: those in which three obtuse interaxial angles may be chosen (Fig. 4) and those in which only two of the angles may be obtuse (Fig. 3), although three acute angles may be chosen. Donnay (1943) has pointed out that statements based on the assumption that it is always possible to select three obtuse interaxial angles have appeared in widely-used texts.

The selection of the morphological unit was formerly the cause of much thought and discussion among crystallographers and resulted in much attention being directed towards form development and crystal habit. Today the structural unit is the basis of almost all morphological descriptions and therefore the units are geometrically identical. Since the use of x-rays has become so common in mineralogy and the selection of the proper structural unit almost routine, the rules for selecting the morphological unit have lost much of their significance. However, studies of the form development of triclinic crystals have a practical application; frequently one of the edges of the conventional structural cell is parallel to the most prominent zone in a crystal or is roughly normal to the broad face of a tabular crystal. Peacock (1937a) has demonstrated that it is often possible to select the conventional structural unit from the gnomonic projection of a well-developed crystal if one of the cell edges can be located in the crystal. The problem is to recognize points of the first layer of the reciprocal lattice in the gnomonic projection of the known forms.

The unit cell is conventionally oriented so that the *c* axis is vertical and the *a* axis is directly fore and aft. This results in (010) being projected directly to the right in gnomonic and stereographic projections.

The Setting of Triclinic Crystals

The literature which deals with the setting of a triclinic crystal, that is with the designation of crystallographic axes and angles within the cell is far from clear-cut and is apt to be confusing to the casual crystallographer. It may be of value to briefly review some of the proposals and rules that have received much prominence.

Donnay, Tunell & Barth (1934) regarded the selection of the "main zone" (to be designated the *c*-axis) of first importance in the problem of selecting the proper morphological setting or orientation. This zone was taken as the direction of elongation (in prismatic or acicular crystals) or perpendicular to the plane of flattening (tabular habit). They suggested that the *a* axis slope forward and the *b* axis slope to the right (which is equivalent to α and β obtuse and implies that the base slopes forward and to the right of the projection of (100)) and that *a* be taken shorter than *b*. The authors pointed out that these rules although arbitrary, were actually the conventions practiced by most crystallographers.

Peacock (1937a) discussed the Donnay-Tunell-Barth morphological setting at some length and named it the normal setting. He extended the idea of the "main zone" to include the prominent edge of a habitually elongated crystal tabular to a plane in the elongated zone. In redefining the steps leading to this setting he limited the slope of the base to the front-right quadrant. This condition was defined alternately as $\phi001$ between 0° and 90°, and α and β obtuse. Later (Peacock, 1937b) the inconsistent statement, α and β obtuse, was discarded, but still later (Donnay, 1943, p. 319) he returned to the original Donnay-Tunell-Barth rule: α and β obtuse.[1]

Buerger (1942) proposed rules leading to a unique setting which entirely disregard the morphology of crystals in favor of purely geometrical relationships within the structural cell. He advocated labelling the three shortest translations so that $a<b<c$ and choosing the positive ends of the axes so as to make α, β and γ all obtuse. These rules are applicable only to a cell of the type shown in Fig. 4 and are therefore, not of general use.

Donnay (1943) pointed out (as did Peacock, 1937a) that the "main zone" is often an edge of the conventional structural unit; in the case of minerals elongated parallel to this zone, the edge is frequently the shortest lattice period. Since the direction of elongation in prismatic crystals is usually taken as the c axis in triclinic crystals, the arrangement $c<a<b$ has long been a common convention. Donnay further pointed out that the concept of the "main zone" lacked clarity; it had been defined as a zone of elongation, as the normal to a broad face, as a zone rich in faces, etc. Donnay therefore proposed to abandon the rule to set the axis of the "main zone" vertical in favour of designating the shortest axis c, but retained the conditions a less than b, and α and β obtuse. In effect Donnay has forged the most common conventions into an inflexible rule. Donnay recognized the recurrence of certain special problems (unusual habit, pseudosymmetry, etc.) which might make this rule unsatisfactory. He therefore agreed to relax his rule to the extent of permitting a cyclic permutation limited to three alternatives: (1) $c<a<b$, α and β obtuse; (2) $a<b<c$, β and γ obtuse; (3) $b<c<a$, γ and α obtuse.

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[1] In Dana (1944, p. 6) the normal setting with the slope of the base to the front-right quadrant is favoured.

The proposals which have been reviewed above are of two types: (1) those which emphasize a consideration of the crystal habit and form development (Donnay-Tunell-Barth, 1934; Peacock, 1937a) and (2) those based on geometrical considerations (Buerger, 1942; Donnay, 1943). It has already been pointed out that Donnay's rules are an attempt to compromise between these two extreme views.

An examination of triclinic settings published in the last 15 years shows that two customs, α and β obtuse, and $a<b$ continue to be much used whether the approach to the problem be from the morphological or the geometrical angle. These customs now have almost the status of rules.

No one convention, with respect to the length of c relative to a and b, is as clearly defined. The Donnay-Tunell-Barth convention regarding designation of the main zone as the c axis is still popular. It has already been mentioned that in the case of triclinic prismatic crystals this often results in the setting $c<a<b$. Contributing to its frequency is the fact that this setting is also selected at times without reference to crystal morphology. The setting $a<c<b$ is not uncommon and results from considerations which are morphological.

Practicing crystallographers frequently choose a setting for the purpose of emphasizing pseudosymmetry, morphological analogy, structural similarity, unusual crystal habit or form development, etc. The resulting crystallographic elements may deviate from the conventional.

Rules based on a consideration of the morphology of crystals cannot lead to a unique setting in all cases. The morphological approach is based on the concept that a mineral has a typical habit and that one of the edges of the conventional cell is parallel to the "main zone." It is probably true that most minerals have an average habit; if a sufficient number of crystals from a variety of localities are examined, certain morphological characteristics will appear. It is probable that a unique setting could be attained for a majority of minerals if the characteristic crystal habit and form development were known. However, first descriptions of crystals are often based on a single specimen from one locality and the morphological development may be quite unusual. Furthermore, the concept of the "main zone" is often open to more than one interpretation on a crystal. For example, the authors first selected the $m\wedge c$ edge on vandenbrandeite as the "main zone" because crystals are elongated parallel to this edge and because it is the zone containing most of the large faces, one of which is the cleavage direction. Our next choice for the "main zone" was the "correct" one: a direction roughly normal to the broad face, containing again the cleavage, and smaller although perhaps some better-developed faces.

Rules that are geometrical have the advantage of leading to a unique setting if the unit is correctly determined by x-rays. However this advantage does not loom as large today. The problem of attaining the proper crystallographic elements was a matter of great concern to geometrical crystallographers of the past. Once the symmetry had been determined, this problem could be resolved into two parts: the selection of the morphological unit, and its orientation in some pleasing or useful way. The first task was the real crux of the problem. That no single straight-forward solution could be found is evident from the various principles and "laws" that were put forward for the guidance of crystallographers. This problem no longer exists for the majority of minerals. The need to find a unique setting from a study of crystal habit and form development is gone because the selection of the conventional structural cell (which is geometrically equivalent to the morphological unit) is now a routine matter with proper x-ray equipment and is controlled by accepted conventions. Today practically all morphological descriptions are based on and accompanied by x-ray data on the conventional unit cell. Although this cell may be oriented in 24 ways, its dimensions are always the same and its angles can differ only by becoming supplementary to those of another orientation. It is therefore easily recognized in contrast to the morphological unit in which the dimensions are given as axial ratios and not readily recognized in another orientation. Reorientation of the cell to another setting for the purpose of a general systematic treatment is a simple straightforward matter. It is important to recognize that the difficult portion of the problem has been solved for us by modern x-ray instruments. At the same time we should view the remaining task, namely the orientation of the cell, in its proper perspective, and realize that a unique setting is not as significant as it was in the past but is a matter that should be decided largely by the needs of the problem in hand.

It is evident then that the selection of the conventional unit cell is the most significant point in choosing the setting. Only in unusual circumstances (as in the plagioclases) is it advisable to depart from this convention. The orientation of this cell should be strongly influenced by the special cases of pseudosymmetry, structural analogy, etc. if noteworthy. It is the representation and discussion of these points that enriches mineralogical literature.[1] In the event that no special features are to be emphasized, the setting should aim at being useful to those who study crystals on optical and x-ray goniometers. To the majority of crystallographers this will continue to mean the selection of some direction in the crystal as the "main zone" and its designation as the c axis. The well established conventions α and β obtuse, and $a<b$ should be observed. Even in special cases it is usually advisable to retain at least the custom α and β obtuse if possible. These commonly used conventions are suggested in order to introduce as much uniformity as possible.[2] If no special direction can be singled out, or if a geometrical scheme is to be followed, the choice of c less than a and b is probably the best because it is in accord with the commonest setting.

APPENDIX

The interpretation of reciprocal lattice projections as plotted from Weissenberg layer photographs is complicated when one of the interaxial angles, either reciprocal or direct, is 90°, or when the negative end of the crystal has been plotted. This is particularly true when morphological information is poorly defined or incomplete. In such cases it may be difficult to determine by inspection, the relative values of the angles with respect to 90° and to locate the positive ends of the axes. It is essential that these circumstances be known before calculation of the direct cell elements can be undertaken. Vandenbrandeite is a special case in that the angle γ^* was measured as $90°0'$. As a matter of interest we explored the possible combinations of angles for conventional triclinic cells in which the angles α and β are never acute. This information may be of value to others and therefore is concisely presented here with the aid of a few sketches (Figs. 3–13). In the reciprocal lattice sketches the centre of the projection is shown as a cross. Direct axes are designated a, b and c, while reciprocal axes are shown as H, K and L.

---

[1] Geometrical rules which do not permit perfect freedom of orientation obviously cannot give adequate expression to these important details.

[2] Donnay's cyclic permutations are unfortunate because they permit α and β acute, and $b<a$ for the mere sake of having a system.

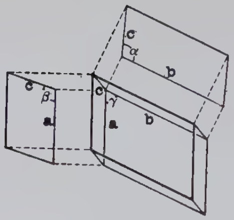

FIG. 3

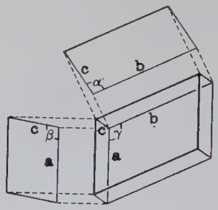

FIG. 4

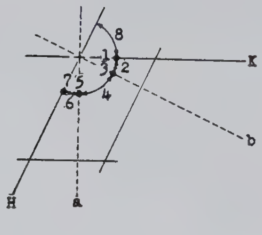

FIG. 5

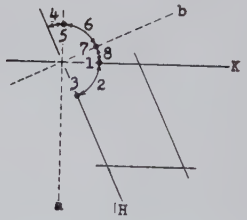

FIG. 6

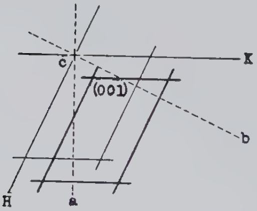

FIG. 7

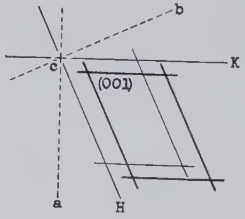

FIG. 8

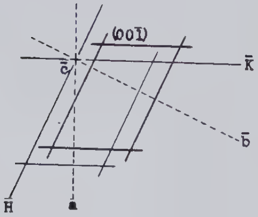

FIG. 9

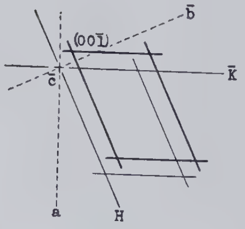

FIG. 10

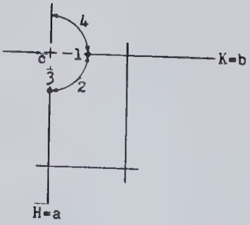

FIG. 11

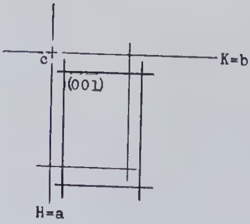

FIG. 12

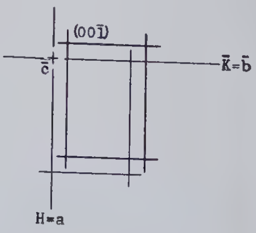

FIG. 13

Figs. 3 and 4 show the two types of oblique triclinic cells. In each case the orientation is chosen so that α and β are obtuse, but in Fig. 3 γ is acute while in Fig. 4 it is obtuse.

In Fig. 5 is represented the *hk*0 layer of the reciprocal lattice for the case in which γ is acute. The numbers 1 to 7 indicate the various positions in which the projection of (001) may fall; for example the reciprocal lattice projection for position 4 is shown in Fig. 7. In position 8 we are dealing with the projection of $(00\bar{1})$, so that c, K and b are negative and γ is obtuse. This is illustrated in Figure 9. The other possible positions of (001) or $(00\bar{1})$ are equivalent to these already indicated and may be brought into coincidence with them by a rotation of 180°. The values of both the reciprocal angles and the direct angles with respect to 90° for each of the 8 positions of (001) of $(00\bar{1})$ are given below.

Position:	1	2	3	4	5	6	7	8
α^*	A[1]	A	A	A	90°	O	O	A
β^*	O	O	90°	A	A	A	A	A
γ^*	O	O	O	O	O	O	O	A
α	O	O	O	O	O	O	90°	O
β	90°	O	O	O	O	O	O	O
γ	A	A	A	A	A	A	A	O

[1] A indicates acute, O obtuse angle.

In Fig. 6 is shown the corresponding diagram for the case in which γ is obtuse. Positions 1, 2 and 3 represent the possible locations of (001), while positions 4 to 8 inclusive represent the positions of $(00\bar{1})$. In the latter locations c, b and K are negative and γ is acute. Figs 8 and 10 illustrate further the angular relations resulting from positions 2 and 6. The relationships of reciprocal angles and direct angles for each of these 8 positions of (001) or $(00\bar{1})$ are given below.

Position:	1	2	3	4	5	6	7	8
α^*	A	A	A	O	90°	A	A	A
β^*	A	A	A	A	A	A	90°	O
γ^*	A	A	A	O	O	O	O	O
α	O	O	90°	O	O	O	O	O
β	90°	O	O	O	O	O	O	O
γ	O	O	O	A	A	A	A	A

Finally in Fig. 11, the case of $\gamma^* = 90°$ has been considered. If the projection of (001) falls in position 1 or 3, there are two right angles in the unit cell and it is geometrically monoclinic. In position 2 (Fig. 12) the direct cell has 3 oblique angles and in position 4 (Fig. 13) the same applies except that here we are looking at the negative end of the crystal. The angular relations are as follows:

Position 2		Position 4	
α^*—acute	α—obtuse	α^*—acute	α—obtuse
β^*—acute	β—obtuse	β^*—acute	β—obtuse
γ^*—90°	γ—acute	γ^*—90°	γ—acute

ACKNOWLEDGMENTS

This work was aided by a scholarship from the Research Council of Ontario (to I. H. M.) and a grant from the National Research Council (to E. W. N.). Mrs. Geraldine M. Killin kindly measured and projected several of the x-ray films.

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STUDIES OF RADIOACTIVE COMPOUNDS:

II—META-ZEUNERITE, URANOPHANE, KASOLITE AND CUPROSKLODOWSKITE IN CANADA[1]

D. D. Hogarth,[2] *University of Toronto*

During the summer of 1950, the writer was engaged in development work on the radioactive prospects of Nicholson Mines Limited whose property is located on the north shore of Lake Athabaska, Saskatchewan. The "No. 1 zone extension" vein which is on the west side of Anne Lake, about 2 miles east of the town of Goldfields, showed evidence of considerable oxidation at surface. It offered an opportunity to study the field relations of secondary uranium minerals in this area and to collect specimens of these little known minerals for subsequent laboratory study at the University of Toronto. The following is a brief account of four rare uranium minerals identified, their mode of occurrence in the vein and its geological environment.

The rocks in the Nicholson region belong to the Tazin series of Precambrian age. In general, the Tazin sediments include dolomite and argillite and their metamorphic equivalents, as well as blue-white and ferruginous quartzite. At the Nicholson an important structural control for radioactive material seems to be the contact between altered dolomite and ferruginous quartzite; the "No. 1 extension" seems to have this relationship.

The vein material is well fractured and weathered. In one place 6 feet of this "soft" rock was excavated with pick and shovel alone. Later shaft sinking showed that this soft material persisted to a depth of more than 40 feet. The vein is at least 40 feet long and has a maximum width of 9 feet. In appearance the vein material resembles the common gossan found above sulphide deposits; it consists chiefly of limonite and quartz.

Mineralogically the vein shows evidence of hydrothermal origin at moderate temperatures. Some 20 minerals were positively identified and there were indications of many more. The paragenesis is uncertain, but in general the following sequence may be applicable:

1. Hematite, sulphides, arsenides.
2. Pitchblende.
3. Quartz.
4. Carbonates.
5. Silicates of uranium, arsenates of uranium.

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[1] Extracted from a thesis for the degree of B.A.Sc. in Mining Geology, *University of Toronto*.

[2] Final Year student in Mining Geology.

This seems to be in accordance with the paragenesis of the Goldfields area as given by Robinson (1950). The sulphides are in masses and locally stain the limonite dark, while malachite, aragonite and bright-colored uranium oxidation products are often seen lining vugs and veinlets. The uranium minerals are of special interest and are described in greater detail below.

Meta-zeunerite, $CuO\cdot 2UO_3\cdot As_2O_5\cdot 12H_2O$, occurs as small green plates (to $\frac{1}{4}$ mm. diameter) in vugs and crevices to the east of the high grade area, in close proximity to sulphides, silver and altered pitchblende. Under the microscope the surfaces of the plates show traces of 2 cleavages at right angles. Cu and U were determined by blowpipe and wet tests. An *x*-ray powder pattern of the mineral was identical with that of synthetic meta-zeunerite prepared by I. H. Milne in this laboratory. This is the first recorded Canadian occurrence for meta-zeunerite.

Uranophane $CaU_2Si_2O_{11}\cdot 6H_2O$, occurs as clusters of radiating tiny yellow to greenish yellow needles lining crevices and as yellow colloform crusts filling cracks in a gum-like uranium mineral. These determinations were made by comparing the *x*-ray powder patterns with well known standards. It appears likely that much of the yellow to greenish yellow stain which contains uranium is uranophane, and this mineral may well be the most common oxidation product of pitchblende here. Uranophane has been reported from three other Canadian localities: Villeneuve, Quebec (Hoffman, 1899); Henvey township, Parry Sound district, Ontario (Spence, 1930); Great Bear Lake (Palache and Berman, 1933). This is the first occurrence identified by *x*-rays.

Kasolite $PbUSiO_6\cdot nH_2O$, was noted as orange yellow crusts in association with an unidentified yellow-green radioactive mineral in fractures near the centre of radioactivity. Both minerals are colloform; kasolite appears to be the later of the two. Microscopic fragments show a prismatic outline. Optically kasolite shows a bluish interference colour; the main index of refraction is above 1.78. Identification was made by comparison of the *x*-ray powder pattern with that of crystallized kasolite from Kasolo, Katanga. The mineral appears to be common in the more highly radioactive areas in the vein. Previous to its identification at Nicholson kasolite was known only from Kasolo in the Belgian Congo.

Of particular interest is the identification of cuprosklodowskite $CuU_2Si_2O_{11}\cdot 6H_2O$, one of the rarest of uranium oxidation products. Previous to its identification at Nicholson this mineral had been found only at Kalongwe, Katanga (Vaes, 1933) and Jachymov, Bohemia (Nováček, 1935). It occurs as tiny bright yellow-green needles in a fissure in talcose argillaceous rock associated with other uranium minerals. It was also seen close to sulphides and malachite on a fine-grained carbonaceous rock as a coating which under high magnification sometimes shows a radiating fibrous structure, and it was noted on the gum-like radioactive mineral mentioned above. Positive tests were obtained for U and Cu and identification was made by comparison with an *x*-ray powder pattern of cuprosklodowskite from Jachymov, Bohemia (HMM-94862).

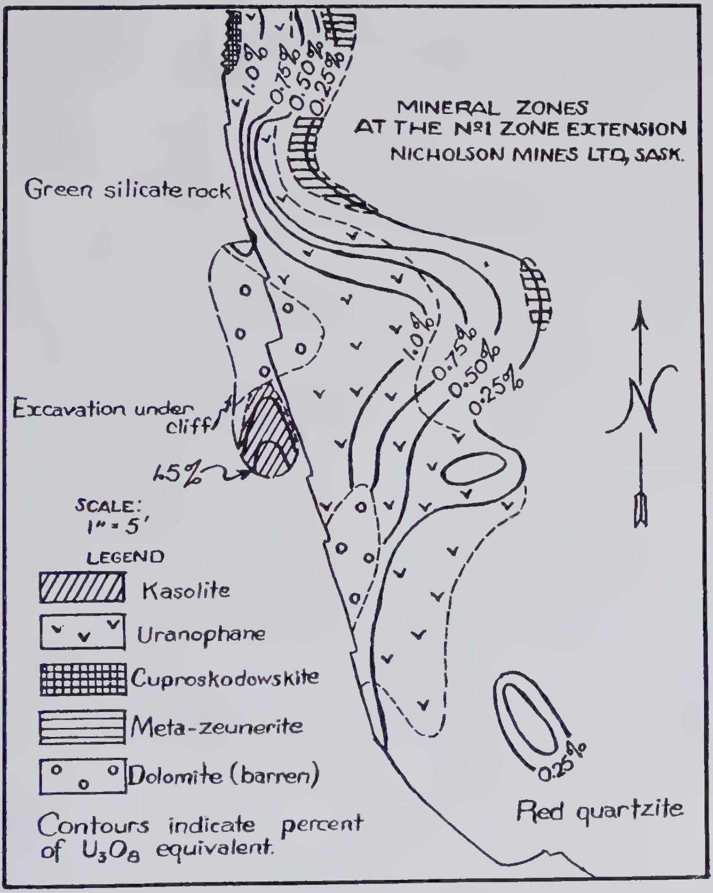

FIG. 1

The accompanying sketch (Fig. 1) is a generalization of the mineral zones as they appear at the Nicholson "No. 1 extension" vein. The grade at the surface (July, 1949) has been roughly blocked out with the aid of contours. It is interesting to compare the mineralogy here with that of uranium occurrences elsewhere. It may be seen that kasolite occurs near the zone of highest uranium concentration. Uranophane and cuprosklodowskite are scattered through a zone of lesser radioactivity. The uranium mica, meta-zeunerite, is distributed at the periphery. This assemblage is similar to that of Shinkolobwe, Katanga. Other analogies are apparent. In contrast to Bohemia where carbonates and sulphates of uranium predominate, silicates of uranium are the principal oxidation products at Nicholson and Katanga. Further, the Cu/Ni+Co ratios are high at both localities. As at Katanga the precious metals Au and Pt are present in significant amounts. Mineralogically therefore, the vein shows distinct similarities to the uranium occurrences in Katanga.

Acknowledgments

The writer is indebted to Professor E. W. Nuffield for assistance and supervision during the study and the preparation of this manuscript. Identification of the uranium minerals was made possible by comparison with standard *x*-ray powder patterns provided by graduate students D. H. Gorman and I. H. Milne. Mr. C. H. E. Stewart of Nicholson Mines Limited kindly gave permission to publish the Company information included in this report.

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STUDIES OF RADIOACTIVE COMPOUNDS:
III—URANO-URANIC OXIDE (U_3O_8)[1]

I. H. MILNE,[2] *University of Toronto*

ABSTRACT

Weissenberg films of U_3O_8 crystals prove the existence of two hexagonal cells whose axes of reference coincide in direction; dimensions $a=3.93$, $c=4.14$kX (cell contents 1/3[U_3O_8]) and $a=3.86$, $c=4.14$kX. Powder photographs indicate the large cell is always present. It is suggested that the "two cell" phenomenon is due to the introduction of oxygen into a portion of the structure. This oxygen may be either in a state of solid solution or in chemical combination necessitating the change of some U^{4+} to U^{6+}.

The compound U_3O_8 is well known in the field of chemistry and has been variously called, including urano-uranic oxide, uranosoic oxide and the green oxide of uranium. It is described (Friend, 1926) as an amorphous powder varying in colour according to its mode of preparation from green to black; the streak is always green. According to both Friend (1926) and Mellor (1932) the compound has not been observed in crystalline form. It is insoluble in water and in dilute acids but in concentrated mineral acids it dissolves slowly forming a mixture of uranous and uranyl salts. The specific gravity is given by Mellor (1932) as 7.31 and by Grønvold (1948) as 8.34.

Urano-uranic oxide is reduced to UO_2 by heating in hydrogen. It is formed when any of the uranium oxides or hydrated oxides or any of the uranium salts with a volatile base or acid is calcined at a red heat (Mellor, 1932). Thus it is readily produced upon heating many uranium-bearing minerals. During the present study of uranium minerals the *x*-ray powder pattern of U_3O_8 was repeatedly obtained from heating experiments. An investigation of the uranium compounds available in the chemical laboratory of this department revealed a quantity of U_3O_8 in the form of a crystalline powder. This enabled the writer to undertake the single crystal study described below.

The origin of the material used is not known. It is a black powder which under high magnification is seen to consist of laths approximately 0.1 mm. in length, showing several prismatic faces. The size of the crystals made it impossible to carry out morphological measurements but it was possible to orient them on the *x*-ray goniometer for rotation about the axis of elongation.

A series of sharp rotation, zero and first layer Weissenberg *x*-ray photographs about the axis of elongation was obtained from one of the laths. These photographs clearly illustrate hexagonal symmetry and indicate the Laue class 6/*mmm*. Measurements gave the following dimensions of the hexagonal unit cell:

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[1] Extracted from a Ph.D. thesis.

[2] Research Council of Ontario scholar.

$$a=3.93\text{kX}$$
$$c=4.14$$

There are no systematic extinctions with the possible exception of a condition in $(000l)$. Because of the extremely small dimensions of the crystals they could not be rotated about a direction normal to the c axis to observe these reflections. The compound therefore, is provisionally placed in the maximum symmetry space group *C6mmm*, of the Laue class 6/*mmm*.

The Weissenberg photographs are complicated by the presence of a complete set of extra reflections which may be interpreted as belonging to a second cell with slightly smaller a dimensions ($a=3.86$kX). These reflections on the single crystal photographs are generally weaker than those of the larger cell but some exceptions were observed. They appear on powder photographs as well and hence cannot be due to twinning. The two cells if such is the case, are so oriented that the c axes coincide in direction and length while the a axes coincide in direction but differ slightly in length. Suggestions as to the origin of this "two cell" structure are given later in this paper.

Grønvold (1948) determined the unit cell of U_3O_8 as orthorhombic base-centered with the following dimensions:

$$a=6.703, \qquad b=3.969, \qquad c=4.136\text{kX}$$

This determination was made from x-ray powder photographs only and the presence of diffraction lines from the two cells resulted in this orthorhombic interpretation. The diffraction lines of the powder pattern can be made to fit Grønvold's orthorhombic cell by taking the smaller $d(10\bar{1}0)$ spacing as $d(200)$ and the larger one as $d(110)$ of the orthorhombic cell. In the same way all of the other lines may be made to fit the orthorhombic cell approximately. However the single crystal photographs obtained during this study leave no doubt of the hexagonal symmetry of urano-uranic oxide.

The powder pattern of the material used for single crystal measurements showed the effects of preferred orientation due to the shape of the crystals. For measurement purposes a more general pattern was obtained from a sample of U_3O_8 supplied by Eldorado Mining and Refining (1944) Ltd. This pattern has been measured and indexed on the basis of the presence of two hexagonal cells. The information is presented in Table 1, and the photograph itself is reproduced in Fig. 1. In this case the diffraction lines indexed on the small cell are in general stronger than

TABLE 1. URANO-URANIC OXIDE (U_3O_8): *X*-RAY POWDER DATA[1]

Hexagonal—*C6mmm:* $a=3.93$, $c=4.14$kX (large cell)
$a=3.86$, $c=4.14$kX (small cell)

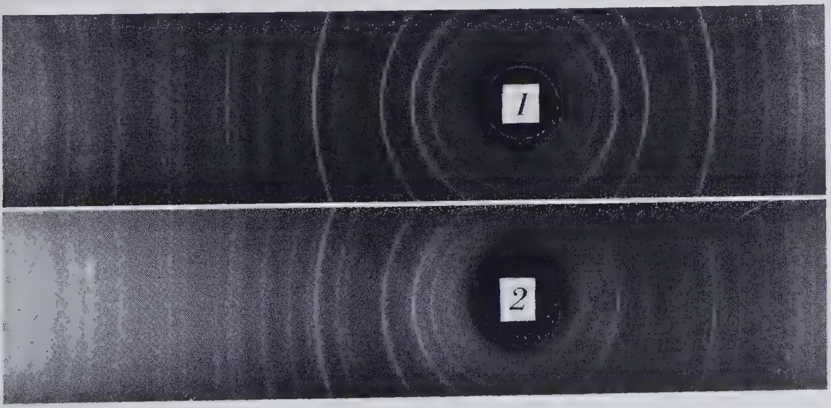
I (Cu)	*d* (meas.)	(*hkil*)	*d* (calc.)	*I* (Cu)	*d* (meas.)	(*hkil*)	*d* (calc.)
4	4.10kX	(0001)	4.14kX	2	1.431kX	$(11\bar{2}2)$	1.425kX
10	3.38	$(10\bar{1}0)$	3.40	3	1.415	$(11\bar{2}2)$*	1.412
7	2.61	$(10\bar{1}1)$	2.65	1	1.376	(0003)	1.380
3	2.06	(0002)	2.07	2	1.314	$(20\bar{2}2)$	1.323
5	1.971	$(11\bar{2}0)$	1.965	4	1.297	$(20\bar{2}2)$*	1.300
6	1.943	$(11\bar{2}0)$*	1.930	5	1.277	$(21\bar{3}0)$	1.286
2	1.782	$(11\bar{2}1)$	1.775	4	1.238	$(21\bar{3}1)$	1.228
8	1.760	$(11\bar{2}1)$*	1.749	3	1.219	$(21\bar{3}1)$*	1.202
5	1.702	$(20\bar{2}0)$	1.700	1	1.134	$(30\bar{3}0)$	1.133
4	1.673	$(20\bar{2}0)$*	1.670	2	1.123	$(11\bar{2}3)$	1.129
4	1.578	$(20\bar{2}1)$	1.574	5	1.100	$(30\bar{3}1)$	1.904
3	1.547	$(20\bar{2}1)$*	1.550			$(21\bar{3}1)$	1.093
$\frac{1}{2}$	1.501	—	—	5	0.991	$(30\bar{3}2)$	0.995

Note—Planes marked with an asterisk are indexed and spacings calculated on the small cell.

[1] For Ni filtered Cu radiation ($\lambda=1.5374$kX).

those of the large cell. This relationship was found in powder patterns of all samples of U_3O_8 which had been subjected to a high temperature.

In Fig. 2 is shown the powder diffraction pattern obtained from the product of heating uranyl nitrate at 220° C. in air for several days. This



FIGS. 1, 2.—*X*-ray powder photographs with Cu/Ni radiation ($\lambda=1.5374$kX); camera radius 57.3 mm.; actual size prints. Fig. 1. U_3O_8, "two cell" pattern. Fig. 2. U_3O_8, single cell pattern.

pattern shows only the lines of the large cell of U_3O_8 and is the only example obtained of a simple structure.

A chemical analysis of this oxide of uranium was carried out using standard methods of analysis. The analysis gave the following results:

U_3O_8	99.0%
Fe_2O_3, V_2O_4	0.4
Total	99.4%

A calculation of the specific gravity of U_3O_8 using the formula $G = MW \times 1.65/V$, where MW is the molecular weight of U_3O_8 and V is the volume of the larger cell gives the value 24.74. This is roughly 3 times the value reported by Mellor (1932) and Grønvold (1948) and it is evident at once that the unit cell derived in this investigation contains only $\frac{1}{3}$ U_3O_8. Grønvold's orthorhombic cell contains $\frac{2}{3}$ U_3O_8; he has suggested that this can be explained by a deficiency structure with some oxygen missing from an ideal UO_3 composition, or with interstitial oxygen in a UO_2 structure. The weakness in this explanation lies in the fact that the compounds UO_2 and UO_3 have *x*-ray powder patterns distinctly different from that of U_3O_8. A more reasonable suggestion in the opinion of the writer is that the *c* dimension of the hexagonal cell is actually 3 times as long as the measured value; the measured value is probably a strong pseudo-period resulting from the arrangement of the uranium atoms in the structure. This problem will be solved only when larger crystals of U_3O_8 are produced so that Weissenberg layer photographs about the *a* axis may be taken.

Experimental observations by Biltz & Müller (1927), and by Alberman & Anderson (1949) on the oxides of uranium have enabled the writer to put forward two suggestions as to the origin of the "two cell" structure of U_3O_8. Biltz & Müller (1927) obtained the dissociation isotherms between 438 and 650° C. of preparations varying in composition from UO_3 to U_3O_8 and found them to be continuous; they also found that the *x*-ray powder patterns of these preparations show the lattice of U_3O_8. They concluded that the solid phase in these experiments was a solid solution. Further experiments were carried out by measuring the densities of uranium oxides (at 25°C.) between $UO_{1.992}$ and $UO_{3.069}$. They state that between UO_2 and $UO_{2.226}$ oxygen is taken up without any change in volume of the oxide and that the composition $UO_{2.226}$ corresponds with the solution of urano-uranic oxide in the dioxide. (Evidently at this point the U_3O_8 pattern appears in *x*-ray powder patterns of the preparations.) Beyond this point the volume increases regularly with no discontinuity at the composition U_3O_8 ($UO_{2.66}$). From these experiments, it may be concluded that solid solution effects are important in determining the composition of the urano-uranic oxide structure. It is possible that the "two cell" structure is due to solid solution effects (with oxygen presumably) which have enlarged the a dimension of the unit cell. In accordance with this suggestion most specimens of urano-uranic oxide must contain two phases, one with more and one with less O than is indicated by the formula U_3O_8; the solution effect giving rise to the higher oxide may be confined to the outer boundaries of grains or crystals where solution of O is most likely. Thus the x-ray diffraction pictures would show the two cells. The U_3O_8 pattern obtained as the result of heating uranyl nitrate at 220° C., which is that of the large cell only can be explained as the result of the formation of urano-uranic oxide under highly oxidizing conditions (decomposition of a nitrate) which would encourage maximum solid solution of oxygen throughout the compound.

More recently, Alberman & Anderson (1949) have made an exhaustive study of the oxygen-uranium system between UO_2 and $UO_{2.3}$ and have found a somewhat analogous double cell phenomenon. In brief, they found that two distinct cubic oxides exist in this range; these are UO_2 (α phase, $a=5.457$kX) and $UO_{2.19}$ (β phase, $a=5.430$kX). The two components appear when oxides in this range of composition are annealed at high temperature. The explanation they offer is that oxygen enters the available interstitial positions in UO_2 in which it may be accommodated in 6-fold co-ordination with uranium, and with very little distortion of the structure. This introduction of oxygen atoms necessitates the change of a corresponding number of U^4 cations to the smaller U^6 cations. The net effect on the cell dimensions is determined by the interplay of sizes and charges of the substituted cations with the distortion caused by the interstitial anions. In this case the shrinkage effect due to the smaller U^6 cations is greater than the expansion effect of the interstitial oxygen anions and therefore the resultant cell is smaller. It is possible that an effect similar to that outlined above is taking place in U_3O_8 and thereby giving the "two cell" structure. If this explanation is correct it would suggest that most U_3O_8 preparations are a mixture of two similar uranium oxide structures with greater and less oxygen than the resultant U_3O_8.

The two suggestions offered for the "two cell" effect are similar in that both depend upon the introduction of O into the urano-uranic oxide structure. Biltz & Müller (1927) feel that this causes an increase in the cell size. Alberman & Anderson (1949) on the other hand have shown that in the case of the oxides UO_2 and $UO_{2.19}$, the latter has a smaller cell volume resulting from the change of valence of U^4 to U^6 necessitated by the introduction of O.

Further experimental work is necessary to arrive at the explanation of the "two cell" phenomenon. It is hoped that the additional information presented here will enable others to carry on the investigation.

ACKNOWLEDGMENT

The writer is grateful to Professor E. W. Nuffield for valuable suggestions during the course of the work and in the preparation of this paper for publication.

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THE EFFECT OF URALITIZATION UPON THE CHEMICAL COMPOSITION OF THE SUDBURY NORITE[1]

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Abstract

Chemical and mineral analyses of fresh and altered norites from Creighton township, and chemical analyses of the constituent ferromagnesian minerals, hypersthene, diallage, and uralite, show that little chemical change except hydration has taken place during the uralitization of the pyroxenes. The results also prove that a Rosiwal analysis of one thin section is not, in general, representative of the mineral composition of a medium-grained rock.

The Sudbury norite as the world's largest and most important nickel intrusive, has been the object of much chemical and mineralogical investigation, a large proportion of it carried out to provide evidence in the classical controversy over the theory of origin of the valuable ore deposits. Although most investigators recognized the severity of the widespread alteration in this rock, few apparently tried to obtain uniformly fresh or altered samples for analysis. This paper gives the results of an attempt to determine whether the process causing the commonest mineral change, that of the pyroxenes to fibrous amphibole—the "uralitization"—has materially affected the chemical composition of the rock and if so, in what way and to what extent. The solution of this problem may lead to a fuller understanding of the mechanism of uralitization in basic rocks in general, and may in particular provide a basis for evaluatng the worth of previous analyses of Sudbury norite specimens, and consequently of the theories of origin based upon these analyses. In addition, it may be possible to establish how carefully sampling must be done on this and similar intrusives to be truly representative.

The general nature of uralitization. The term "uralite" was first applied by Rose (1812) to a mineral with the crystalline form of a pyroxene and the structure of an amphibole. Dana (1892) states that the change begins on the outer surface of the pyroxene transforming it into an aggregation of slender amphibole prisms, parallel in position to each other and the parent pyroxene. When complete, the entire crystal is changed into a bundle of fibers. The most prominent changes in composition are those in lime and magnesia. Clarke (1924, p. 600) points out that the change is complicated in many cases by the simultaneous formation of epidote or zoisite. A separation of magnetite may occur and calcite may be formed due to the removal of lime. Generally augite alters to hornblende or actinolite, and diopside to tremolite.

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[1] Extracted from an unpublished M.Sc. thesis of the same title—*University of Manitoba*, 1949. This work was carried out with the assistance of a bursary from the National Research Council (Canada) with whose permission this paper is published.

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The causes of uralitization have received only occasional attention. Duparc & Hornung (1904) suggested that, after the pyroxene had crystallized in the magma, it was acted on by the residual magma and the transformation occurred into patches of amphibole. In a later paper, Duparc (1908) said that the phenomenon of uralitization is confined chiefly to feldspathic rocks and he attributed it to a slightly later local injection of feldspathic magma. He cites cases in the Asiatic Urals where uralitization is caused by the injection of leucocratic dikes into pyroxenites. The common occurrence of uralitization as a deuteric alteration effect has been noted by Colony (1923). In the end stages of crystallization some of the quartz and the alkalies are concentrated in liquid form of low viscosity and great penetrating power. At this stage olivine and magnesia-rich pyroxenes are susceptible to alteration resulting in serpentinization or uralitization, the latter showing a powerful penetrating power by invading the feldspar in every direction. He also notes that these phenomena may alter the chemical and mineral composition of the rock. Schwartz (1939) stated that uralitization is a frequent product of hydrothermal alteration, but that it may occur during metamorphism of various types.

Previous analyses of the Sudbury norite and deductions from them. The first important investigation was carried out by Walker (1897) who analysed five samples from across the intrusive in the neighbourhood of the Blezard mine, and from the results concluded that there is a regular increase in acidity towards the centre of the intrusive. No other complete series of analyses was made until Knight (1923) analyzed seventeen samples from the intrusive in MacLennan township. From these he concluded that there was no gradual increase in acidity; in fact, "the most basic part of the eruptive is not along the outside, but from 1,500 to 2,100 feet from the outside edge." Phemister (1925) attacked the problem in a different manner by doing mineral analyses on samples from eleven traverses across the intrusive. He concluded that the norite of the south range is most basic from one-half to three-quarters of its total width of outcrop from the basal contact. Above this, it becomes more acid by the development of a mesostasis of quartz and alkali feldspar and beneath it by the presence of quartz, which is a late crystallization. Coleman, Moore, & Walker (1929), dissatisfied with the above and other conclusions, chemically analyzed samples from across four complete sections of the intrusive: the Capreol and Levack sections on the north range, and the Cameron Mine and Creighton on the south. Their conclusion was "that the eruptive sheet is a unit formed by the segregation of a magma into a more basic portion, norite, passing without a break upwards into a more acid phase, micropegmatite."

A point which all the above investigators appear to have overlooked is that the deuteric alteration, particularly the uralitization, may have considerably altered the bulk composition of the rock. Suppose, for example, that one exposure of fresh norite and another in which all the pyroxene and part of the feldspar have been altered to amphibole are found twenty feet apart, then which exposure should be sampled, or does it make any difference? If the samples are chosen at random, they will be in different stages of alteration, and may not give a true picture of the composition gradient. It may be necessary to obtain samples which are at least fairly uniformly altered.

Some of the discrepancies which *may* be due to this late alteration are found in the previously published analyses. For example, an analysis of a sample from forty-seven yards northwest of the Creighton mine gives only 0.69% MgO (Knight, 1917); this can hardly be typical norite. Another analysis of Knight's of a sample from 1,083 yards northwest of Creighton shows 1.56% MgO, but analyses of two samples straddling this one (Coleman *et al.*, 1929) show 5.51 and 6.62% MgO respectively.

Mineral analyses, too, may not be representative unless special precautions are taken to make them so, for the change in mode can be great without appreciably affecting the chemical composition. The method itself may introduce large errors as Larsen & Miller (1935) have emphasized: "Certain it is that many rocks, particularly coarse-grained rocks, are not well mixed on the scale represented by a thin section." This statement certainly applies to the norite in which the ferromagnesians have a strong tendency to cluster. Larsen & Miller found that Rosiwal analyses of thin sections from the same hand specimen varied by from 2 to 10% for the major constituents, and they warn that "the danger of basing far-reaching conclusions on a few Rosiwal determinations of specimens from widely scattered outcrops is evident." Phemister's work, however, cannot be criticized in this regard because all his results are shown by graphs, and there is no account of the number of samples or thin sections that he used.

EXPERIMENTAL RESULTS

Samples. The norite samples used in the present study were collected on Concessions 1, 2 and 3, Lot 2, Creighton township, about the centre of the norite zone. They were taken from below the zone of weathering and thus could not have been subjected to surface alteration.

General petrography. Unaltered Sudbury norite is composed essentially of plagioclase, hypersthene, diallage, and quartz or granophyre. The order of crystallization of these minerals is clearly shown by the textural relationships. Hypersthene was generally the first mineral to crystallize although in some sections it is later than plagioclase, indicating that the two minerals crystallized in part contemporaneously. The irregular shape of diallage and its habit of enclosing euhedral crystals of hypersthene and feldspar show that the diallage is later. Quartz, or a granophyric intergrowth of quartz and soda feldspar, or rarely microcline, was the last important mineral to crystallize, as shown by its habit of filling in between the larger minerals in characteristic triangular or irregular shapes, and by its corrosion of the adjacent feldspar.

Plagioclase occurs as euhedral laths of a distinctive brown color. It is sodic labradorite. The brown color of the feldspar is due to minute inclusions of ilmenite, hematite, or magnetite as indicated by an analysis reported by Coleman *et al.* (1929) showing it to contain 0.23% TiO_2 and 1.10% FeO. In places these inclusions can be seen in the plagioclase as minute black needles in small patches, forming a nearly right-angled grid structure. Hypersthene occurs in euhedral stubby prisms which are commonly clustered together, and has the refractive indices $\alpha = 1.692$, and $\gamma = 1.699$ (both ± 0.002). Diallage is present as large irregular grains, commonly as simple twins, and has the indices of refraction $\alpha = 1.689$, $\gamma = 1.710$ (± 0.002). Other primary minerals are hornblende, magnetite, apatite, and yellow sulphides. Primary hornblende is of minor occurrence, and is distinguished from secondary amphibole by its blue-green color and non-fibrous nature. The magnetite and apatite show evidences of both early and late crystallization, and they also show highly variable amounts in different sections, but these special problems were not investigated during the present study.

Alteration. The nature of the uralitic alteration is observed more easily in the hypersthene than in the diallage which has a lamellar structure controlling the amount and distribution of the fibrous material. In hypersthene the alteration always begins at the outer border and works progressively inwards by veining until only isolated remnants of the fresh pyroxene remain; in diallage, because of the structural control, most of the mineral may be altered and yet have partly fresh borders. Both hypersthene and diallage may alter directly to uralite or may pass through a bastitic phase with the separation of small grains of magnetite. Serpentine, the identification of which was confirmed by an x-ray powder photograph, is gray to light green in color and has a massive or aggregate structure. The uralite is pale gray to green in color with indices of refraction $\beta = 1.631$, $\gamma' = 1.643$ (± 0.002). As alteration intensifies, the uralite recrystallizes around the outer edges to a darker green, more pleochroic amphibole, and it may become mixed with many small biotite plates. With extreme alteration, this mixture or the uralite alone spreads out through the rock replacing the feldspar along grain boundaries and twinning planes or in irregular patches.

Alteration of the plagioclase is by no means as common or intense as that of the pyroxenes, but even in the freshest norite an incipient type of alteration occurs at the centre of the large feldspar grains. Rarely this alteration is intense and the large feldspar crystal loses its brown color and becomes packed with small crystals which resemble zoisite. With severe alteration, the feldspar may break down completely, lose its brown color, and the rock becomes a mixture of clear albite, epidote, and amphibole.

TABLE 1. CHEMICAL ANALYSES OF FRESH AND ALTERED NORITE

Constituents	Fresh	Altered
SiO_2	53.30	54.42
Al_2O_3	17.60	19.65
Fe_2O_3	2.88	1.87
FeO	7.13	6.07
CaO	9.05	8.74
MgO	6.06	5.15
Na_2O	2.58	2.80
K_2O	1.34	1.53
H_2O+	0.22	0.30
Total	100.16	100.53
S. G.	2.87	2.88

Analyses by T. A. Oliver.

The results in table 1 show that there is little chemical difference between fresh and altered norite; the differences are so small that they may be within the range of experimental error. Silica and alumina appear to be added in the altered sample, whereas there is an apparent decrease of all other constituents except the alkalies and water. These differences are slight, however, and may not be significant. The water content of both samples is surprisingly low, but redeterminations gave similar results.

Separation and analysis of hypersthene, diallage, and uralite. The differences in composition of the fresh and altered rock are small, but possibly some of the differences are real. To determine whether or not this is so, pure hypersthene, diallage, and uralite were separated from crushed rock and analysed. Two samples about fifteen feet apart, one fresh and the other altered, were chosen for the separation. Pure uralite was obtained with ease by separating the minus 35-plus 60 fraction in Clerici's solution. The first concentrate of uralite was purified by two further separations. By counting grains in index oil, the concentrate was found to be 97% pure. The separation of hypersthene and diallage was complicated by the fact that the specific gravity of diallage, about 3.28, is intermediate between that of hypersthene and uralite, about 3.37 and 3.04 respectively. Therefore, hypersthene partially altered to uralite has about the same specific gravity as the diallage, and these come down together in the separation. By repeated separations, however, about 1.5 grams of 98% pure hypersthene was recovered from the mixture, and about half a gram of 95% pure diallage was laboriously picked by hand under the binocular microscope.

Table 2. Chemical Analyses of Hypersthene, Diallage, and Uralite

Constituents	1	2	3	4
SiO_2	52.11	51.61	52.34	54.3
Al_2O_3	1.22	4.17	5.01	
Fe_2O_3	1.65		2.26	18.4
FeO	19.96	11.48	13.04	
CaO	1.63	16.09	6.98	8.4
MgO	22.16	15.49	15.06	18.1
K_2O			0.80	0.8
H_2O+			0.71	
Total	98.73	98.84	96.20	100.0

1. Analysis of hypersthene, close to $Mg_2FeSi_3O_9$.
2. Analysis of diallage, close to $Ca_2(Mg, Fe)_4Si_6O_{18}$.
3. Average of two analyses of uralite.
4. Average of uralite recalculated to 100% by taking into account addition of silica +alumina and water (including the deficit).

Analyses by T. A. Oliver.

The results of the chemical analyses of these three minerals are shown in table 2, along with the closely approximated chemical formulae of hypersthene and diallage. Only the main portion of these two minerals was completed, owing to the small amounts available. The analysis of uralite shows it to be roughly intermediate in composition between hypersthene and diallage. The first analysis of uralite showed a deficit of about 4%. This was considered to be an analytical error but a second analysis verified the first. The nature of this analytical deficit in amphibole has been discussed by Jakob (1937), who found that it is generally absent from asbestiform (one-dimensional) varieties but never from compact (three-dimensional) varieties. The deficit was once thought to be due to an excess of "molecular" oxygen but Jakob has shown and others have confirmed that it is actually caused by water given off above 1000° C. This water is more or less reduced to hydrogen by divalent titanium.

Calculations based on the analyses. It is now possible, by making simple calculations involving the compositions of hypersthene and diallage, and the ratio of hypersthene to diallage, to determine whether the uralitization was accompanied by any change in chemical composition. The composition of uralite theoretically calculated from the composition and proportions of hypersthene and diallage should agree with the composition of uralite found by chemical analysis if no changes in chemical composition accompany the uralitization. The ratio of hypersthene to diallage in the rock was determined by Rosiwal analyses of 13 thin sections and by several grain counts to be about 1.7 to 1, which means that 0.63 parts hypersthene plus 0.37 parts diallage form one part uralite.

Several assumptions and adjustments have to be made before making the calculations. If it is assumed that none of the constituents change except silica+alumina and water, then the other constituents must be adjusted to 100% before a true comparison can be made. The assumption that there is an actual gain in silica+alumina agrees with the results of the rock analyses, in which there was a total gain of about 3% in these constituents in the altered rock. In the uralite there is a gain of about 3.1% in silica+alumina assuming a hypersthene: diallage ratio of 1.7:1, and a gain of 4.5% in water assuming the whole deficit to be water, making a total gain of 7.6%. This amount is divided proportionally among total iron, lime, and magnesia in the uralite should be equivalent to those in the two pyroxenes when the ratio of these two minerals in the norite is taken into account if the above assumptions are valid. Using the above hypersthene:diallage ratio, uralite theoretically derived from hypersthene and diallage should contain 18.0% total iron whereas analyzed uralite adjusted to 100% contains 18.4% ferrous iron. Theoretically, the uralite derived solely from the hypersthene and diallage should contain 6.9% lime and 19.6% magnesia; actually it contains 8.4% lime and 18.1% magnesia.

If the above calculations are absolutely correct, it means that in the uralite formed from the pyroxenes by alteration, 0.4% total iron and 1.5% lime have been added, and 1.5% magnesia has been lost. However, when consideration is given the numerous sources of possible error, the separation of pure minerals, the chemical analyses themselves, and the determination of the hypersthene to diallage ratio, the differences are probably within the limits of error, and the results appear to indicate no marked change in composition with the process of uralitization.

Nature of the uralite. The secondary amphibole in the norite may have a constant composition or two distinct amphiboles may have been formed from the two pyroxenes. If there are two, then the hypersthene and diallage merely underwent hydration in the deuteric alteration, but if there is only one of constant composition then there must have been an interchange of calcium, magnesium, and iron. The optical properties do not afford much evidence. The uralite formed from hypersthene does appear different from that formed from diallage in slightly altered specimens, that derived from diallage having higher interference colors and being finer-grained than that derived from hypersthene. However, the lamellar structure of diallage may control the size of the alteration product. Further evidence suggesting two amphiboles is that the hypersthene seems to be much more easily altered than the diallage; some sections show much of the hypersthene severely altered whereas much of the diallage is still fresh. In severely altered norite, all of the uralite has the same appearance, suggesting a constant composition.

As mentioned earlier, hypersthene occurs in euhedral prisms whereas diallage has an irregular habit. In the early stages of alteration, the secondary amphibole retains these distinctive shapes. It was therefore possible to polish a section of rock and scrape out, with a diamond point, enough of the two types for *x*-ray powder photographs. Again the results were inconclusive; the data from both patterns are in substantial agreement with each other and with those given in the A.S.T.M. Card Index (1945) for actinolite (II–1908). Thus, no conclusion is possible concerning the comparative chemical compositions of the two uralites, the one derived from hypersthene, the other from diallage.

CONCLUSIONS

The conclusions based on the above work are as follows:

(1) The results of chemical analyses of fresh and altered norite show that, in the samples investigated, no important changes in the chemical composition of the rock occur as a result of the process causing uralitization. This fact was confirmed by chemically analysing hypersthene, diallage, and uralite separately; these results show that water and possibly a small amount of silica and alumina are added in the formation of uralite whereas the differences in the other constituents are within the limits of experimental error. However, no samples of norite were analysed in which the feldspar had been largely replaced by the uralite or late quartz, and such material may be radically altered in composition. Furthermore, there may have been a change in bulk composition due to the process of uralitization in other parts of the intrusive.

(2) A Rosiwal analysis of one thin section will not in general give reliable values for the mineral composition of a medium or coarse-grained rock.

(3) The process of uralitization in the Sudbury norite appears to be carried out by a residual liquid, possibly carrying small amounts of silica and alumina. In the first stages of the alteration, simple hydration of the pyroxene takes place, resulting in serpentinization or uralitization of the hypersthene and leaving the diallage relatively unaltered. With increasing alteration an interchange of calcium, magnesium, and iron between the two pyroxenes may occur, resulting in the formation of an amphibole of constant composition.

ACKNOWLEDGMENTS

The writer wishes to gratefully acknowledge the help of Dr. H. D. B. Wilson of the Department of Geology, University of Manitoba, who conceived the problem and directed the work. Helpful criticism was received from the other members of the Department, Dr. G. M. Brownell, Mr. E. I. Leith, Mr. G. A. Russell, and from Dr. R. B. Ferguson who assisted in the preparation of this paper for publication. Dr. Cordell Durrell, of the University of California, Los Angeles, also offered valued assistance and advice.

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FURTHER NOTES ON THE PRECISION OF THE BUERGER PRECESSION INSTRUMENT

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Abstract

Tests of various factors that might affect the accuracy of unit cell constants obtained from Buerger precession films are described. The use of horizontal and vertical fiducial dots for evaluating film shrinkage is advocated.

During the summer of 1949 this laboratory commenced using fiducial dots on zero level precession films for estimating film shrinkage. Since then a number of tests of various factors that might affect the accuracy of measurement of precession films have been carried out and a summary is presented herewith. The instrument in use was supplied by Mr. Charles Supper in 1948 and has the variable F.

Film Shrinkage and Fiducial Dots

Provision is made in the Supper instrument for imprinting two dots on the x-ray negative in the zero level cassette on a horizontal line through the centre of the film, parallel to the dial axis. These fiducial marks establish a horizontal axis for purposes of crystal orientation from angular measurements carried out on the precession films (Evans, Tilden & Adams, 1949). In connection with a redetermination of the unit cell constants of sucrose in this laboratory, A. W. Hanson (1949) used the distance apart of these dots on the film as compared with the distance apart of the holes in the back plate of the zero level cassette to determine film shrinkage. These tests have now been repeated and extended to film shrinkage measurements along the vertical direction as well as the horizontal.

The distance apart of the fiducial dots on a film when the film shrinkage is zero was established as follows. The distance between the pin-holes in the back plate of the zero level cassette on the side in contact with the x-ray film was measured by means of a travelling microscope. The scale of the Buerger precession measuring device (Buerger, 1945), (Charles Supper Co.), having a vernier reading to 0.05 mm., was compared with that of the travelling microscope by measuring the distances between the fiducial dots of several exposed and processed x-ray films on both instruments. As a further check, several exposures of the pin-holes were made on Kodak Studio Proof (printing out) paper, replacing x-ray film in the cassette but requiring no wet processing, and the distances apart of the dots on the paper were measured with the precession measuring device. It probably is advisable to determine the distance apart of the dots for zero film shrinkage separately for each instrument. In the instrument employed for the present tests a new back was made to improve the light-tightness of the zero level cassette. The two holes for imprinting the dots are 11.725 cm. apart which brings them just within the illuminated square area of the measuring device. The distance apart of the holes in the cassette of a new precession instrument recently received, however, is 12.214 cm. which places the dots over the flange supporting the clear glass plate of the viewer. In this laboratory about $\frac{1}{2}$ inch has been removed from the centre of each side of the flange to illuminate the area around the horizontal and vertical pairs of dots on the films.

The following procedure is recommended for the determination of film shrinkage corrections to be applied to measurements on zero level precession photographs. Immediately after the x-ray exposure, the horizontal pair of pin-holes is exposed for a few seconds to ordinary light in the darkroom. (It is desirable that a standard set of conditions be set up in order that the spots on each film shall be sufficiently well defined, without halation and of the same intensity from film to film.) The square back of the cassette is then lifted out, turned through 90° and replaced. The pin-holes, now vertical, are again exposed to light and the film is removed and processed. The horizontal pair of fiducial dots on the film continues to serve its original purpose of establishing a horizontal line; the vertical pair, however, is not normally used to define a vertical line because of the possibility of slight disturbance of the position of the film in the cassette when the back is removed, turned and replaced. In actual practice, however, it has been found that the vertical dots define a line that is seldom more than 5 or 10 minutes from being strictly perpendicular to that joining the centres of the horizontal dots.

Using Kodak No-Screen X-ray Film, size 5 in. × 7 in., cut to 5 in. × 5 in. for the zero level cassette, the film shrinkage in the seven-inch direction has always differed from that in the five-inch direction regardless of which direction was vertical or horizontal during tank processing. Although exhaustive tests have not been made, a survey of some sixty films representing six boxes (75 sheet size) and four film lot numbers suggests that the direction of maximum shrinkage is associated primarily with the lot number, usually being the same for different boxes of the same lot. In two boxes of the same lot number, however, the film shrinkage was greater in the five-inch direction throughout one box and more than half-way through the second. It then changed to the seven-inch direction and this persisted to the end of the box. Presumably the reason is to be found in the manufacturing process or cutting routine at the factory.

In magnitude the film shrinkage commonly is of the order of 0.15% to 0.45% in the direction of larger shrinkage although it has been observed as high as 0.9%. In the direction of smaller shrinkage it usually amounts to 0.05% to 0.15% although values up to 0.3% have been encountered. The difference between the shrinkage in the two directions on a given film frequently is less than 0.3% but may be as high as 0.5% on occasion. To take full advantage of the precession instrument, therefore, in the measurement of unit cell constants, both horizontal and vertical fiducial dots should be printed on the film before processing and film shrinkage corrections should be evaluated separately for the two directions on the film.

No appreciable change (less than 80 microns) was found in the distances between the fiducial spots on two *x*-ray films measured on the travelling microscope at intervals over a period of two months. During times of extreme change in humidity or temperature, however, the apparent film shrinkage may change by significant amounts and even negative shrinkage corrections are possible.

Horizontal Axis, Vertical Axis and Goniometric Arcs

The line joining the horizontal fiducial dots may be tested for parallelism with the rotation axis of the goniometer head as follows. By means of a template the plane of one of the arcs is brought parallel to the plane of the film in the zero level cassette and the dial reading is noted. For convenience the set-screw in the dial may be loosened and the dial turned to a reading of 0°00′ and the set-screw tightened. A crystal, such as quartz, is mounted and, by means of precession setting photographs, orientated by means of the arc parallel to the plate so that a principal reciprocal lattice line is exactly collinear with the horizontal fiducial dots. The dial is turned through exactly 180° from this position and another precession photograph is taken. If the fiducial dots are parallel with the rotation axis the reciprocal lattice line will again be collinear with the fiducial dots. If they are not, the crystal should be moved on the arc by one-half the angle required to remove the difference. The test may be checked using the other arc and dial positions 90° and 270° with reference to the first position. The line joining the fiducial dots in the present instrument is off parallelism with the rotation axis by an angle of less than 5 minutes.

Based on this test, a most convenient method of completing the accurate setting of a crystal, already advocated by Evans, Tilden & Adams (1949), is to bring a reciprocal lattice line truly horizontal (*i.e.*, along the dial rotation axis) at dial settings 0° and 90° (alternate arcs parallel to the film) and then check it by photographs at 180° and 270°. That leaves only a dial correction necessary to make the required zero level perpendicular to the *x*-ray beam.

A series of tests of the effect of missetting of the dial on measurements of Fd^* was made. Two series, each of eleven *c*-axis zero level photographs of a crystal of childrenite with b^* vertical, were recorded over a range of 50 minutes (dial reading) at positions 180° apart and similar series with a^* vertical. A set of *c*-axis zero level photographs of quartz with a^* horizontal also was obtained covering a 4° range. The results showed (a) a small difference in dial readings between those photographs in which the criterion for perfect setting was the white radiation "tails" from the outermost spots to the limit of the zero level circle, and those having equal distances from the outermost diffraction spots to the center of the level, and (b) slight differences in Fd^* apparently dependent on dial missetting. The observed differences may be fortuitous but the important feature of these tests was to demonstrate that missetting of the dial by as much as 2° does not affect the accuracy of measurement of Fd^* by more than about 0.3%.

The detailed results of these tests indicated that the magnitude of Fd^* obtained for an axis set horizontally might not be identical with that observed when the same axis was vertical. As a test of this point and of the overall accuracy of the precession instrument under average conditions, a series of photographs of a quartz crystal was taken. The prismatic crystal (from a miarolitic cavity in a quartz cobble, Pitman Field Beach, Marblehead, Mass.) was clear and well formed, about 0.37 mm. long and 0.15 mm. in cross-section. It was mounted with the a^*, (orthohexagonal), b^* and c^* axes, respectively, along the horizontal (dial) axis of the instrument. Using molybdenum radiation six photographs were obtained (a^* horizontal, b^* vertical; a^* horizontal, c^* vertical; b^* horizontal, a^* vertical b^* horizontal, c^* vertical; c^* horizontal, a^* vertical; c^* horizontal, b^* vertical). This series was then repeated using copper radiation. Measurements of Fd_a^*, Fd_b^*, Fd_c^* were employed to calculate F which had been set as closely as possible at 6.00 cm. For this purpose the following data were chosen: values of a and c given by Wilson and Lipson (1941) ($a=4.90320$ kX, $c=5.39371$ kX), converted to international Ångström units by the ratio of the wavelength of copper $K\alpha_1$, 1.54050 Å to that used by Wilson and Lipson (1941), 1.537395 kX, giving $a=4.9131$ Å, $c=5.4046$ Å, $b(=\sqrt{3}a)=8.5098$ Å. In each case horizontal and vertical measurements on the films were corrected for film shrinkage determined directly for the horizontal and vertical directions from the fiducial dots on the films. Results are shown in Table 1. In this table the second, third and fourth columns give the values of F obtained from Fd^* measurements along the designated (a^*, b^*, c^*) axes when the latter were horizontal; the last three columns give the F values calculated from Fd^* measurements along the same axes when they were vertical.

TABLE 1. F (CMS.)

Radiation	Horizontal			Vertical		
($K\alpha$)	a^*	b^*	c^*	a^*	b^*	c^*
Mo	6.000	6.003	6.003	5.981	6.002	5.990
Mo	5.988	5.997	6.008	5.985	5.998	5.998
Cu	6.007	6.011	6.013	6.001	5.989	5.992
Cu	6.001	6.001	5.998	5.982	6.000	5.978

It must be emphasized that the films were of average quality only, some better than others, and no attempt was made to obtain an ideal photograph in every case. In general, those obtained with copper were not as easy to measure as those with molybdenum, partly due to greater absorption by the crystal and partly due to the smaller number of row lines on the films. The object, however, was to test the instrument under average, rather than ideal, conditions so all films were included in the results.

In each of these films, shrinkage was greater in the vertical direction. Without correction for film shrinkage all values of F calculated from Fd^* measurements made along the vertical axis were lower (5.959 cm. to 5.980 cm.) than those obtained from Fd^* measurements along the horizontal axis (5.983 cm. to 6.008 cm.). The average of all values of F after correcting for film shrinkage (Table 1) is 6.002 cm. $\pm 0.2\%$ based on horizontal axes and 5.992 cm. $\pm 0.2\%$ based on vertical axes. This confirmed the indication that experimental values for direct lattice constants might not be the same when the axis was horizontal as when it was vertical. It seems desirable, therefore, to investigate this point for each instrument[1] and, if necessary, to use two values for F depending on whether a given axis under investigation is horizontal or vertical. Alternatively, a single F value may be used and the difference in the "apparent" F's can be incorporated in the distances apart of the horizontal and vertical fiducial dots for zero film shrinkage. That the difference in the present instrument, although small, probably is real receives support from the fact that in all twelve quartz films the corrected F value from the horizontal measurements was greater than that obtained from the vertical measurements in every case except one. Some earlier photographs of quartz and of eosphorite also showed the same small difference between measurements of Fd^* when a given axis was vertical and horizontal, respectively.

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[1] *Note added in proof.* Values of F obtained with a new precession instrument, having a fixed F, are 5.994 cm. $\pm 0.06\%$ for horizontal measurements and 5.997 cm. $\pm 0.15\%$ for vertical measurements, with a mean for all values of 5.995 cm. $\pm 0.15\%$, *i.e.*, in this case there is no significant difference. The older instrument, therefore, may be slightly out of adjustment.

The personal factor in measuring precession films was tested by having two people independently measure some of the foregoing films. Resulting values for F usually differed by less than 0.1% and never by more than 0.25% even for the poorer films.

Various methods of averaging the measurements of row line spacings were tested but resulting values of Fd^* did not differ by more than about 0.1%.

Small missettings of the goniometer arcs of 0.1° and 0.2° and miscentering of the crystal within the limits of the collimator aperture did not affect measurements of Fd^* by more than 0.1%.

Conclusions

H. T. Evans (1949) has recognised the importance of film shrinkage on the accuracy with which unit cell dimensions can be obtained with the precession camera. The use of vertical and horizontal fiducial dots allows a film shrinkage correction to be applied. Experience in this laboratory indicates that provided film shrinkage is taken into account, that the F setting is calibrated against a suitable standard such as quartz, and that reasonable care is exercised in orientating the crystal, the maximum probable error in unit cell constants determined with the precession instrument should not exceed ±0.3% and should seldom be more than ±0.2%. Previous estimates (Barnes, 1949; Evans, 1949) of about ±0.25% for the accuracy to be expected from the precession instrument under normal routine operating conditions thus appear to be fully justified.

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MATILDITE, ARAMAYOITE, MIARGYRITE[1]

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ABSTRACT

Matildite, aramayoite, and miargyrite are mineral examples of non-isomorphous structures, the atomic arrangements in which are nevertheless closely similar. Triclinic face-centred sub-cells with similar dimensions to the face-centred cubic cells of the high temperature isomorphous series $AgBiS_2$-$AgSbS_2$ can be chosen in all three structures, and are manifest in the galena-like grouping of the powder pattern lines. Long annealing of the metastable artificial members below the inversion temperature induced partial ordering in their disordered structures and gave products with similar powder patterns to those of matildite and aramayoite. New orthorhombic body-centred lattice dimensions, $a=3.918$, $b=4.046$, $c=5.662$ kX are given for matildite; the structural lattice dimensions of aramayoite are refined to Triclinic Pinacoidal-$\bar{1}$, $a=7.72$, $b=8.82$, $c=8.30$ kX, $\alpha=100°22\frac{1}{2}'$, $\beta=90°00'$, $\gamma=103°54'$; and indexed powder patterns are given for all three minerals and three of the annealed artificial products.

A series of seven pyrosynthetic compounds covering the full range $AgBiS_2$-$AgSbS_2$ gave a practically constant face-centred cubic pattern of the galena type. The three known minerals in this series, matildite $AgBiS_2$, aramayoite $Ag(Sb,Bi)S_2$ with $Sb:Bi=5:1$, and miargyrite $AgSbS_2$ are orthorhombic, triclinic, and monoclinic, respectively, with complex x-ray powder patterns in which groups of lines can be recognized in place of single lines in the galena-type patterns of the pyrosynthetic products. From this it is apparent that there are high temperature (cubic) and low temperature (non-cubic) forms throughout the series. Pyrosynthesis produces the high temperature (α) forms which on rapid cooling persist as metastable compounds at room temperature. From the fact that these α-modifications give the galena type of pattern, it is evident that their unit cells contain $Ag_2(Bi,Sb)_2S_4$, with Ag, Bi, and Sb acting as equivalent metallic atoms in a rock-salt arrangement, M_4S_4. In the corresponding minerals representing the low temperature β-modifications the modified galena type of pattern shows that these less symmetrical structures represent some modification of the rock-salt arrangement by atoms of different kinds assuming special positions in larger cells with lower symmetries. This is entirely in keeping with the known disorder-order relations in many metallic compounds.

It was therefore decided to attempt to induce the α-β inversion in artificial compounds of the series, and also to correlate the crystallography of the three minerals which superficially show little relation to their common cubic high temperature prototype.

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[2] Mineragrapher, Department of Mines and Technical Surveys. Ottawa.

Matildite $AgBiS_2$ is commonly more or less intergrown with galena ("schapbachite," "plenargyrite"). Ramdohr (1938) studied these intergrowths in detail with the aid of *x*-ray powder photographs made with a heating camera. Below about 210° C. mineral samples gave mixed patterns of matildite (β-$AgBiS_2$) and galena; above this temperature the pure galena type of pattern was obtained indicating inversion of matildite to α-$AgBiS_2$ which is isostructural with galena and makes a homogeneous solid solution with the lead compound. For α-$AgBiS_2$ Ramdohr gives $a=5.64$ A; β-$AgBiS_2$ was found to be orthorhombic with $a=8.14$, $b=7.87$, $c=5.69$ A, from the powder patterns.

Aramayoite $Ag(Sb,Bi)S_2$ (Spencer, 1926) was studied with the Bragg *x*-ray spectrometer by Yardley—now Mrs. K. Lonsdale—(1926), who obtained a pseudo-cubic triclinic cell with $a=5.67$, $b=5.69$, $c=5.623$ A, $\alpha=86°55'$, $\beta=90°53'$, $\gamma=93°18'$. From the intensities it appeared that the heavy atoms, Ag, (Sb,Bi), must make a face-centred lattice while the light S atoms might be placed as in a rock-salt type of structure. On well developed crystals of aramayoite from the original locality Berman & Wolfe (1939) obtained entirely different triclinic cell dimensions: $a=7.76$, $b=8.79$, $c=8.34$ kX, $\alpha=100°22'$, $\beta=90°00'$, $\gamma=103°54'$. This cell conforms to the morphology but shows no immediate relation to the pseudo-cubic cell of Yardley.

The structure of miargyrite was determined by Hofmann (1938) using the Weissenberg method. The distribution of the strong diffractions on the photographs showed a pseudo-cubic face-centred arrangement of the heavy atoms which was recognized as similar to that in the aramayoite structure given by Yardley, and to the probable structure of matildite. Hofmann obtained monoclinic lattice dimensions, $a=13.17$, $b=4.39$, $c=12.83$ kX, $\beta=98°37\frac{1}{2}'$.

MATILDITE

As the lattice dimensions of matildite given in Ramdohr (1938) had been obtained from powder patterns alone, it was considered desirable to try to prepare single crystals of β-$AgBiS_2$ on which to check the cell elements. Accordingly, crystals giving the face-centred cubic powder pattern of α-$AgBiS_2$ were hydrosynthesized in a pressure bomb from alkaline sodium sulphide solution of appropriate constituents, after the method developed by Dr. F. G. Smith of the University of Toronto. Several dozen of these, ranging in size from 0.1 to 0.5 mm., were picked under the microscope from their locations scattered freely throughout blades and felted masses of crystalline bismuth sulphide, or attached loosely by an edge or apex to masses of anhedral silver sulphide. Microchemical tests showed silver, bismuth, and sulphur. The Talmadge hardness was B+, estimated by needle scratch on an octahedral face. Careful crushing yielded a black powder assuming dark reddish tones on finer pulverization, while rubbing under the pestle revealed some malleability and produced thin gray flakes. Powder photographs on crushed crystals showed a cubic face-centred pattern giving a cell-edge which compares closely with that given by Ramdohr (1938) for matildite above 215° C.; a check on the composition of the hydrosynthetic material was obtained by an identical powder photograph from a homogeneous air-quenched fusion product of composition $AgBiS_2$.

α-$AgBiS_2$ (pyrosynthetic)	$a=5.636$ kX A.R.G.
α-$AgBiS_2$ (hydrosynthetic)	$a=5.635$ kX A.R.G.
α-$AgBiS_2$ (matildite at 215° C.)	$a=5.64$ kX Ramdohr (1938)

Several hydrosynthetic crystals and a portion of the fusion product of the same composition were sealed in separate evacuated pyrex tubes together with a few milligrams of sulphur to combine with residual oxygen. The tubes were heated in a constant temperature oven at 180° C. for nine days, then air-quenched. Part of the fusion product was mounted and polished for optical examination. Weak anisotropism and accentuated grain boundaries were seen upon comparison with the original isotropic material. Etch-tests upon the annealed portion were almost identical with those given by Ramdohr (1938) for matildite, but less rapid action was observed with 1:1 HNO_3.

Powder photographs of the annealed fusion product and of annealed crystals showed each line of the face-centred cubic pattern divided into two or more separate lines, indicating a change to lower symmetry, more complete in the case of the fusion product. In the pattern of the latter material, several of the strong lines of Ag_2S appeared after annealing. Re-examination of the polished section at high magnification revealed tiny areas of Ag_2S and Bi_2S_3, in approximately equal amounts, less than 1% of the whole, included in grains of the main phase to one side of the section. Fortuitous inclusion of Ag_2S in the annealed x-ray powder sample had apparently occurred in an amount large enough to suggest segregation of two phases during annealing, although the amount of uncombined Ag_2S present by visual comparison was insufficient to affect to any noticeable degree the composition of the main phase. Incomplete reaction of the components during fusion is the most likely explanation.

An individual annealed crystal rotated on a random axis in the powder camera gave a pattern which indicated almost complete inversion, and whose spots superimposed perfectly upon all but the Ag_2S lines of the powder pattern of the annealed fusion product. Comparison of the measurements with measurements on a powder pattern of natural matildite and galena from Schapbach, Baden, by R. M. Thompson, showed substantial agreement in the spacings and intensities of lines after elimination of the galena lines from the pattern of the mineral. Thus α-AgBiS$_2$, cubic in symmetry, had inverted to a β-form of lower symmetry identical with matildite.

The same crystal which had yielded the β-AgBiS$_2$ powder pattern was oriented to rotate about the apparent four-fold symmetry axis. The signals on the optical goniometer were weaker and more diffuse than before inversion, but the measured angles were still within a few minutes of those of the octahedron. A rotation photograph about this axis superimposed upon one taken before inversion showed retention of cubic diffractions, much diminished in intensity, and the appearance of extra diffractions from inverted portions of the crystal. Layer lines were paired, and two choices of period were possible, one 5.662 kX; the other 5.50 kX calculated from the spacings of each pair of ($hk1$) and ($hk2$) layer lines registered on the photograph.

Weissenberg zero and first layer photographs about this same axis were moderately successful, but a twelve hour exposure failed to resolve weak second-layer diffractions. The zero layer diffractions were characteristic of a body-centred orthorhombic lattice twinned on (110). Extra diffractions defining the twinned orthorhombic lattice appeared on the ($h00$) and ($0k0$) rows closely bracketing spots which superimposed perfectly on those of the zero layer photograph about the same axis, a principal cubic axis before inversion. The cubic diffractions were assumed to belong to an uninverted portion of the crystal. A schematic representation of the zero and first layers of the twinned reciprocal lattice appears in Fig. 1 with the cubic diffractions omitted. In the terminology of Friedel the obliquity of the twin is 1°50′.

From the Weissenberg and rotation photographs elements were calculated which agree poorly with those given for matildite by Ramdohr (1938), after halving and interchanging his a and b periods.

Matildite (Ramdohr)	β-AgBiS$_2$ (A.R.G.)
$b/2=3.935$ kX	$a=3.918$ kX
$a/2=4.07$	$b=4.046$
$c\ \ =5.69$	$c=5.662$

Table 1 contains the powder pattern of artificial β-AgBiS$_2$ and that of material labelled matildite from Schapbach, (galena lines eliminated) indexed using the elements obtained from the inverted hydrosynthetic crystals and also according to the pseudo-cubic face-centred setting analogous to the high temperature lattice. Better agreement was obtained between measured and calculated spacings than with the elements quoted in Ramdohr (1938).

The relation of the body centred orthorhombic unit cell of β-$AgBiS_2$ (matildite) to the face-centred cubic (now pseudo-cubic) cell of α-$AgBiS_2$ is shown in Figure 2a. One might infer that the inversion α to β was accompanied by a segregation of the (Ag, Bi) atoms, which are equivalent in the cubic structure, into Ag atoms, say, at the origin of the orthorhombic cell and Bi at the body-centre. However, the fact that β-$AgBiS_2$ still gives the systematically missing spectra of the body-centred lattice shows that such segregation of Ag and Bi cannot have proceeded very far.

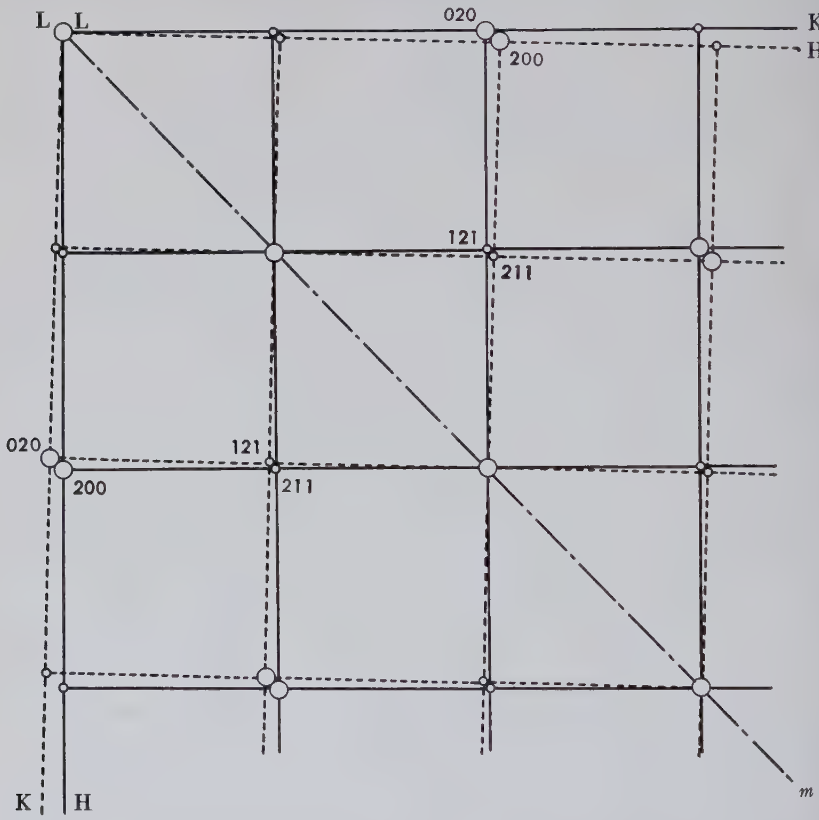


FIG. 1.—Schematic reciprocal lattice plot from 0- and 1-layer Weissenberg photographs of twinned annealed $AgBiS_2$ crystal. Large circles are 0-layer diffractions, small circles 1-layer diffractions. Solid and dotted lines define lattices of individuals twinned on (110). Chained line is the trace of the twin plane $m(110)$.

ARAMAYOITE AND MIARGYRITE

The unit cell of aramayoite was checked by Professor Peacock from a consideration of the results of Berman & Wolfe and those of Yardley to-

TABLE 1. COMPARISON OF MATILDITE AND β-$AgBiS_2$ POWDER PATTERNS

Orthorhombic, I; $a=3.918$, $b=4.046$, $c=5.662$ kX; $Z=1$

1		2		3	4	5
I(Cu)	d(meas.)	I(Cu)	d(meas.)	(hkl)	(hkl)	d(calc.)
6	3.32kX	8	3.29 kX	(111)	(011)	3.292 kX
2	3.18	1	3.16	$(1\bar{1}1)$	(101)	3.222
10	2.83	10	2.82	(002)	(002)	2.831
				(200)	(110)	2.814
3	2.03	6	2.03	(220)	(020)	2.023
5	1.963	5	1.967	$(2\bar{2}0)$	(200)	1.960
3	1.711	3	1.708	(311)	(121)	1.713
				(113)	(013)	1.710
½	1.670	½	1.670	$(3\bar{1}1)$	(211)	1.683
1	1.648	½	1.648	(222)	(022)	1.646
2	1.583	½	1.586	$(2\bar{2}2)$	(202)	1.611
3	1.396	1	1.411	(004)	(004)	1.416
				(400)	(220)	1.407
4	1.322	1	1.316	(331)	(031)	1.312
½	1.285	1	1.282	(420)	(130)	1.275
2	1.249	1	1.247	$(4\bar{2}0)$	(310)	1.243
—	—	½	1.167	(422)	(132)	1.163
½	1.160	½	1.158	(224)	(024)	1.160
—	—	½	1.072	$(5\bar{1}1)$	(321)	1.077
				(333)	(303)	1.074
—	—	½	0.968	(531)	(141)	0.965
—	—	½	0.884	$(6\bar{2}0)$	(420)	0.882

1. Measurements and observed intensities on material from Schapbach, Baden, Germany, with galena lines removed; after R. M. Thompson, University of Toronto (unpublished data).
2. Measurements and observed intensities from annealed artificial crystals, by A. R. Graham (Ag_2S lines removed).
3. Indices according to pseudo-cubic face-centered lattice.
4. Indices in structural setting.
5. Spacings calculated from structural elements.

gether with some new measurements on type material. The adopted cell elements differ only slightly from those of Berman & Wolfe (Table 2) and the correctness of the new values is indicated by the indexed powder pattern (Table 4). The transformation of Yardley's setting to that of Berman & Wolfe was also verified.

TABLE 2. ARAMAYOITE: CELL DIMENSIONS

	1	2	3	4
a	7.72 kX	—	(7.76) kX	—
b	8.82	—	(8.79)	
c	8.30	8.34 kX	(8.34)	—
d(100)	7.49	7.52	7.50	$6d(33\bar{1})$ 7.56 kX
d(010)	8.42	8.41	8.43	*d*(001) 8.415
d(001)	8.16	—	8.16	$6d(\bar{3}31)$ 8.13
α	100°22½′	—	100°22′	—
β	90°00′	90°00′	90°00′	—
γ	103°54′	—	103°54′	—

1. Adopted by Peacock as best representing the available measurements.
2. Measured by Peacock.
3. Given by Berman & Wolfe (1939); *a, b, c,* were computed in a manner which cannot now be ascertained; α, β, γ, are morphological values.
4. Spacings observed by Yardley (1926) on the ionization spectrometer; it is assumed that Yardley's planes $(\bar{3}1\bar{3})$ are in fact $(\bar{3}31)$.

Hofmann's (1938) unit cell of miargyrite was retained and the relation to the pseudo-cubic sublattice was worked out with the help of information in his published work.

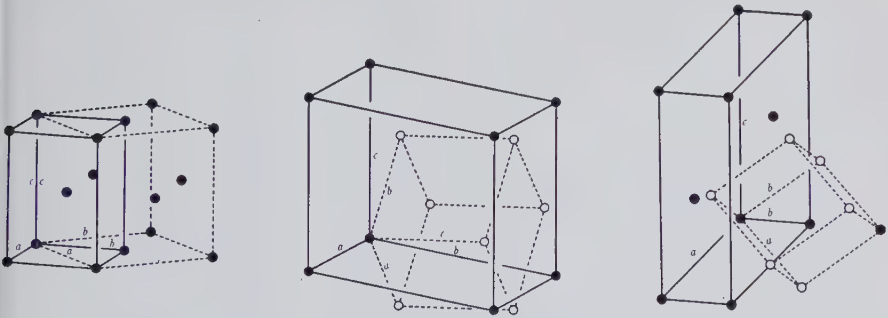
For convenient comparison, the cell dimensions of the pseudo-cubic face-centred sub-lattices present in all three minerals were calculated using the general formulae for lattice periods and angles. The transformation formulae involved in the calculation of cell elements are as follows:

Matildite:	
Graham to Pseudo-cubic	$110/\bar{1}10/001$
Pseudo-cubic to Graham	$\tfrac{1}{2}\overline{\tfrac{1}{2}}0/\tfrac{1}{2}\tfrac{1}{2}0/001$
Aramayoite:	
Berman & Wolfe to Yardley	$\overline{\tfrac{1}{2}}0\overline{\tfrac{1}{2}}/\overline{\tfrac{1}{2}}0\tfrac{1}{2}/\tfrac{1}{6}\tfrac{2}{3}\tfrac{1}{6}$
Yardley to Berman & White	$\bar{1}\bar{1}0/\tfrac{1}{2}0\tfrac{3}{2}/\bar{1}10$
Miargyrite:	
Hofmann to Pseudo-cubic	$\tfrac{1}{8}1\overline{\tfrac{1}{4}}/\overline{\tfrac{1}{8}}1\tfrac{1}{4}/\tfrac{3}{8}0\tfrac{1}{4}$
Pseudo-cubic to Hofmann	$1\bar{1}2/\tfrac{1}{2}\tfrac{1}{2}0/\tfrac{3}{2}\tfrac{3}{2}1$

The dimensions of the sub-lattices given in Table 3 show a striking similarity which is not immediately evident in the lattices established by *x*-ray and optical goniometrical studies. Figs. 2a, b, c show the relationship of the sub-lattices to the lattices in structural settings by means of individual cells viewed in the conventional manner. The pseudo-cubic face-centred cell of β-$AgBiS_2$ is actually twice as large as the unit cell but for the sake of clarity, is still called a sub-lattice by analogy with those of the other two minerals. In the diagrams, structural lattice points are filled circles; sub-lattice points are left open. In all the structures, all these points must be occupied by heavy atoms (Hofmann, 1938).

TABLE 3. COMPARISON OF LATTICE DIMENSIONS (kX)

	Matildite		Aramayoite		Miargyrite	
	Struct. Lattice	Ps-cub. Sub. Latt.	Struct. Lattice	Ps-cub. Sub. Latt.	Struct. Lattice	Ps-cub. Sub. Latt.
a	3.918	5.632	7.72	5.67	13.17	5.818
b	4.046	5.632	8.82	5.67	4.39	5.818
c	5.662	5.662	8.30	5.62	12.83	5.471
α	90°00′	90°00′	100°22½′	86°42′	90°00′	88°58′
β	90°00′	90°00′	90°00′	91°33′	98°37½′	91°02′
γ	90°00′	88°10′	103°54′	94°8½′	90°00′	82°02′
V	89.76	179.5	539.1	179.9	733.4	183.3 kX^3
Z	1	2	6	2	8	2



(a) (b) (c)

FIG. 2.—Structural unit cells (solid lines) and pseudo-cubic sub-lattices (dotted lines) of (a) matildite, (b) aramayoite, (c) miargyrite. Filled circles are structural lattice points; open circles are sub-lattice points. Face-centre points of sub-lattice omitted for clarity in (b) and (c).

It is apparent that an ordering process during inversion (cooling) which involves a slight adjustment of bond length as well as the lowering of symmetry by assumption of special positions by atoms of particular elements, could satisfy the observed facts concerning the lattices of all

TABLE 4. ARAMAYOITE-$Ag(Sb,Bi)S_2$: X-RAY POWDER PATTERN
Triclinic, $\bar{1}$; $a=7.72$, $b=8.82$, $c=8.30$ kX;
$\alpha=100°22\frac{1}{2}'$, $\beta=90°00'$, $\gamma=103°54'$; $Z=6$

I(obs.)	θ(Cu)	d(meas.)	(hkl)*	(hkl)	d(calc.)
2	12.95°	3.43 kX	$(\bar{1}11)$	(012)	3.428 kX
4	13.85	3.21	$(1\bar{1}1)$	$(02\bar{2})$	3.244
			(111)	$(\bar{2}20)$	3.213
1	14.15	3.15	$(\bar{1}\bar{1}1)$	(210)	3.146
10	15.90	2.806	(200)	$(\bar{2}1\bar{2})$	2.826
			(020)	$(\bar{2}02)$	2.822
			(002)	(030)	2.804
2	22.05	2.048	(022)	$(\bar{2}32)$	2.047
$\frac{1}{2}$	22.45	2.015	$(\bar{2}02)$	(222)	2.014
$\frac{1}{2}$	23.00	1.967	(202)	$(\bar{2}4\bar{2})$	1.968
3	23.35	1.940	$(0\bar{2}2)$	$(23\bar{2})$	1.936
			(220)	$(\bar{4}10)$	1.930
$\frac{1}{2}$	25.95	1.757	$(\bar{1}31)$	$(\bar{2}14)$	1.767
			$(\bar{3}11)$	(204)	1.757
1	26.25	1.738	$(\bar{1}13)$	(042)	1.742
2	26.80	1.705	$(3\bar{1}1)$	$(\bar{2}3\bar{4})$	1.716
			$(\bar{2}22)$	(024)	1.714
			$(1\bar{3}1)$	$(22\bar{4})$	1.704
			(113)	$(\bar{2}50)$	1.698
			(131)	$(\bar{4}22)$	1.691
1	27.45	1.668	$(\bar{3}\bar{1}1)$	(402)	1.673
			(311)	$(\bar{4}3\bar{2})$	1.669
			$(1\bar{1}3)$	$(05\bar{2})$	1.668
			$(\bar{1}\bar{1}3)$	(240)	1.668
1	28.45	1.614	$(2\bar{2}2)$	$(04\bar{4})$	1.622
			(222)	$(\bar{4}40)$	1.607
$\frac{1}{2}$	29.15	1.578	$(\bar{2}\bar{2}2)$	(420)	1.573
$\frac{1}{2}$	32.95	1.413	(400)	$(\bar{4}2\bar{4})$	1.413
			(040)	$(\bar{4}04)$	1.411
2	33.25	1.402	(004)	(060)	1.402

I(obs)	θ(Cu)	d(meas.)	I(obs)	θ(Cu)	d(meas.)	I(obs)	θ(Cu)	d(meas.)
$\frac{1}{2}$	34.85°	1.345 kX	$\frac{1}{2}$	37.25°	1.270 kX	$\frac{1}{2}$	42.65°	1.135 kX
$\frac{1}{2}$	36.45	1.294	$\frac{1}{2}$	38.35	1.239	$\frac{1}{2}$	45.05	1.086
$\frac{1}{2}$	36.80	1.283	$\frac{1}{2}$	41.75	1.154	$\frac{1}{2}$	45.85	1.071

* Pseudo-cubic face-centered sub-lattice indices.

TABLE 5. MIARGYRITE-$AgSbS_2$: X-RAY POWDER PATTERN

Monoclinic, $A2/m$; $a=13.17$, $b=4.39$, $c=12.83$ kX; $\beta=98°37\frac{1}{2}'$; $Z=8$

I(obs.)	θ(Cu)	d(meas.)	(hkl)*	(hkl)	d(calc.)
9	12.90°	3.443 kX	(111)	(211)	3.423 kX
2	14.00	3.178	$(\bar{1}11)$	(004)	3.171
2	14.40	3.091	$(1\bar{1}1)$	$(40\bar{2})$	3.091
10	15.50	2.877	(200)	$(21\bar{3})$	2.881
8	16.30	2.739	(002)	(402)	2.734
$\frac{1}{2}$	20.40	2.205	(220)	(020)	2.195
4	22.55	2.005	(022)	(215)	2.004
4	23.10	1.963	$(0\bar{2}2)$	$(6\bar{1}\bar{1})$	1.963
3	23.75	1.909	$(2\bar{2}0)$	$(40\bar{6})$	1.909
1	25.30	1.799	(131)	(024)	1.805
2	25.40	1.792	(311)	$(42\bar{2})$	1.790
$\frac{1}{2}$	26.65	1.714	(222)	(422)	1.712
3	27.20	1.682	(113)	(613)	1.684
			$(\bar{1}31)$	$(\bar{2}17)$	1.679
1	27.65	1.657	$(\bar{1}13)$	(406)	1.663
			$(1\bar{3}1)$	$(6\bar{1}5)$	1.655
$\frac{1}{2}$	28.25	1.624	$(1\bar{1}3)$	(800)	1.628
2	29.00	1.586	$(\bar{2}22)$	(008)	1.585
2	29.85	1.544	$(2\bar{2}2)$	$(80\bar{4})$	1.545
$\frac{1}{2}$	31.10	1.488	—	—	—
$\frac{1}{2}$	32.15	1.445	(400)	$(42\bar{6})$	1.440
$\frac{1}{2}$	32.90	1.415	(331)	(231)	1.414
1	34.20	1.368	(004)	(804)	1.368

I(obs)	θ(Cu)	d(meas.)	I(obs)	θ(Cu)	d(meas.)	I(obs)	θ(Cu)	d(meas.)
$\frac{1}{2}$	35.40°	1.327 kX	$\frac{1}{2}$	43.05°	1.126 kX	1	53.60°	0.955 kX
$\frac{1}{2}$	35.90	1.311	$\frac{1}{2}$	44.05	1.106	$\frac{1}{2}$	56.30	0.924
$\frac{1}{2}$	36.90	1.280	$\frac{1}{2}$	44.35	1.100	$\frac{1}{2}$	58.40	0.902
$\frac{1}{2}$	37.50	1.263	$\frac{1}{2}$	45.10	1.085	$\frac{1}{2}$	59.10	0.896
2	38.10	1.246	$\frac{1}{2}$	45.60	1.076	$\frac{1}{2}$	61.10	0.878
$\frac{1}{2}$	38.70	1.229	$\frac{1}{2}$	46.35	1.062	$\frac{1}{2}$	62.40	0.867
$\frac{1}{2}$	38.90	1.224	$\frac{1}{2}$	50.05	1.003	$\frac{1}{2}$	65.80	0.843
$\frac{1}{2}$	41.40	1.162	$\frac{1}{2}$	51.15	0.987	$\frac{1}{2}$	69.15	0.823
$\frac{1}{2}$	42.25	1.144	$\frac{1}{2}$	52.00	0.975	$\frac{1}{2}$	75.15	0.795

Elements after Hofmann (1938).

* Pseudo-cubic face-centred sub-lattice indices. Measurements by Graham on U. of T. Spec. R569: Miargyrite Braunsdorf, Saxony. Indexed by transformation from pseudo-cubic face-centred sub-cell.

three compounds. As would be expected, the miargyrite sublattice shows the greatest aberrance from a high-symmetry disordered lattice: the smaller radius of the Sb atom compared to those of Ag and Bi would produce a greater distortion.

For the identification of aramayoite and miargyrite their complex *x*-ray powder patterns were indexed in a practical way suggested by Professor Peacock. This is based on the assumption that the powder diffractions would probably all be due to the heavy atoms, which are situated at the points of the face-centred pseudo-cubic sub-lattices; and that therefore it would suffice to calculate the spacings of all sets of planes, in the desired range, corresponding to indices conforming to the *F*-condition in the sub-lattice. This assumption proved to be justified as seen in the indexed patterns (Tables 4 and 5).

INVERSION EXPERIMENTS IN THE SYSTEM $AgBiS_2$-$AgSbS_2$

The successful inversion of the metastable α-$AgBiS_2$ led to an examination of the results of annealing fusion products throughout the system $AgBiS_2$-$AgSbS_2$. Six further fusions including four wtih purposely irrational atomic ratios of Bi to Sb, were carried out in evacuated tubes. The quenched products were examined in polished section and by *x*-ray powder photographs. All appeared entirely homogenous and gave face-centred cubic powder patterns indicating disordered structures metastable at normal temperatures. The compositions and measured cell-edges are given in Table 6. The cell-edges were calculated from back reflections from the planes (046), (155) (117), and (444). The adjustment method employed is considered to have reduced the probable error to ±.003 kX. The fact that the cube-edge is practically constant throughout the series indicates that the large Ag atom in some way controls the lattice parameters in the disordered high temperature structures.

TABLE 6. COMPOSITIONS AND CELL-EDGES OF FUSIONS

Fusion	Composition	Cell-edge
A	$AgBiS_2$	5.636 kX
B	$Ag_{10}Bi_9Sb_1S_{20}$	5.640
C	$Ag_{10}Bi_7Sb_3S_{20}$	5.641
D	$Ag_2Bi_1Sb_1S_4$	5.645
E	$Ag_{10}Bi_3Sb_7S_{20}$	5.642
F	$Ag_{10}Bi_1Sb_9S_{20}$	5.636
G	$AgSbS_2$	5.642

Slow cooling of the fusions over periods up to a few hours in length, had little or no effect on the powder patterns, so the same annealing treatment as with $AgBiS_2$ was attempted upon each of the six new fusions, B to G. Fusions B and F inverted nearly completely after annealing at 180° C. for eight days, while the rest remained either unchanged or showed only slight diffusion of the powder lines indicating some tendency toward inversion. Further treatment at 200° C. for nine days produced definite changes in D and E. Fusions C and G remained unchanged after annealing at 180°, 200°, 210°, 215° C., for periods up to five weeks in duration. After the last annealing experiment attempted, fusion G gave the cubic powder pattern with faint additional extraneous lines which were not obviously related to those of miargyrite or to any of the other low temperature patterns.

Table 7 presents the measured spacings in kX units and the observed intensities of the powder patterns from inverted products, together with portions of the patterns of α-$AgBiS_2$, aramayoite and miargyrite, indexed according to the pseudo-cubic face-centered setting. It will be seen that the inverted product E gives a powder pattern closely comparable to that of aramayoite. An inversion product comparable to miargyrite was not obtained.

Conclusions

The successfully annealed fusion products in the system $AgBiS_2$-$AgSbS_2$ possess pseudo-cubic sub-lattices closely related to one another and to those possessed by the minerals matildite and aramayoite. These sub-lattices are descended from those of the face-centred cubic high temperature forms, which are truly isomorphous and have disordered structures.

True isomorphism cannot be held to exist in the low temperature forms, even although the arrangement of heavy atoms in the structures is much the same, for the ordering process initiated and accelerated by annealing destroys strict isomorphism by unequal shifts in atomic positions according to the availability of Bi or Sb atoms. Consequently, different structures are possible, although the basic structures differ but slightly.

Possibly a number of structures could be produced during the ordering process in a single compound of this type by annealing within appropriate temperature limits for periods sufficient to permit maximum ordering. In nature, extremely slow cooling from temperature of crystallization may be regarded as the optimum condition for the ordering process to proceed to completion during the fall of the temperature level to normal. We should expect changes to have taken place in the symmetry of some minerals which are irreproducible in the laboratory simply from lack of time.

The crystal habits of aramayoite and miargyrite are in complete harmony with their crystal lattices and show no cubic pseudo-symmetry.

TABLE 7. COMPARISON OF MEASURED SPACINGS OF ANNEALED FUSIONS B AND E α-$AgBiS_2$, β-$AgBiS_2$, ARAMAYOITE, MIARGYRITE IN kX UNITS

α-$AgBiS_2$			β-$AgBiS_2$			B		E		Aramayoite			Miargyrite		
I	*d*	*hkl*	*I*	*d*	*hkl*	*I*	*d*	*I*	*d*	*I*	*d*	*hkl*	*I*	*d*	*hkl*
								1	3.39	2	3.43	$\bar{1}11$	9	3.44	111
7	3.23	111	8	3.29	111	1	3.28								
			1	3.16	$1\bar{1}1$	10	3.19	6	3.22	4	3.21	$1\bar{1}1$ 111	2	3.18	$\bar{1}11$
						½	3.16			1	3.15	$\bar{1}\bar{1}1$	2	3.09	$1\bar{1}1$
10	2.81	002	10	2.82	002 200	6	2.84	10	2.81	10	2.81	200 020 002	10	2.88	200
						6	2.75						8	2.74	002
						2	2.72								
8	1.98	022	3	2.03	220	½	2.03	2	2.05	2	2.05	022	½	2.21	220
								1	2.02	½	2.02	$\bar{2}02$	4	2.01	022
			5	1.97	$2\bar{2}0$	5	1.98			½	1.97	202	4	1.96	$0\bar{2}2$
						3	1.93	3 ½	1.94 1.90	3	1.94	$0\bar{2}2$ 220	3	1.91	$2\bar{2}0$
													½	1.80	131
								½	1.76	½	1.76	$\bar{1}31$ $\bar{3}11$	1	1.79	311
4	1.69	113				2	1.72	1	1.75	1	1.74	113			
			3	1.71	311 113	½	1.70	5	1.71	2	1.71	$\bar{3}11$ $\bar{2}22$ $1\bar{3}1$ 113 131	½	1.71	222
			1	1.67	$3\bar{1}1$	1	1.67	2	1.68	1	1.67	$\bar{3}\bar{1}1$ $3\bar{1}1$ $1\bar{1}3$ $\bar{1}\bar{1}3$	3	1.68	113 131
													1	1.66	131 $\bar{1}13$
													½	1.62	$1\bar{1}3$
2	1.63	222	1	1.65	222	1	1.65	1	1.62	1	1.61	$2\bar{2}2$ 222	2	1.54	$\bar{2}22$
			½	1.59	$2\bar{2}2$	2	1.61			½	1.58	$\bar{2}\bar{2}2$	2	1.54	$2\bar{2}2$
								½	1.52		—		½	1.49	—
1	1.41	004	1	1.41	004 400	½	1.43	3	1.41	½	1.41	400 040	½	1.45	400
						½	1.38						½	1.42	331
										2	1.40	004	1	1.37	004

B		E					
I	*d*	*I*	*d*	*I*	*d*	*I*	*d*
½	1.355	½	1.344	½	1.269	½	1.093
½	1.322	½	1.330	½	1.235	½	1.074
½	1.292	½	1.314	½	1.191	½	0.989
1	1.228	½	1.289	½	1.134	½	0.970
½	1.073	½	1.280				
½	0.983						

From this it is almost certain that these minerals crystallized below the inversion temperatures like acanthite and that they are not paramorphs of the corresponding high temperature cubic phases (like argentite).

ACKNOWLEDGMENTS

The investigation summarized in this article forms part of a thesis submitted in conformity with the requirements of the degree of Doctor of Philosophy in the University of Toronto, 1950. It was begun in 1947 during the summer session at Queen's University under the friendly guidance of Professor L. G. Berry, by agreement with the Department of Geological Sciences and the School of Graduate Studies of the University of Toronto. It was pursued at the latter institution during the winter sessions of 1947–48 and 1948–49 with the assistance of two scholarships granted by the Ontario Research Council, and completed and prepared for publication in the Mineragraphic and Spectrographic Laboratories of the Mineral Dressing and Process Metallurgy Division of the Mines Branch, Department of Mines and Technical Surveys, Ottawa.

The author was indebted to Professor M. A. Peacock for his friendly supervision and constructive criticism throughout the course of the work. Professor F. Gordon Smith gave many useful suggestions concerning hydrosynthetic and pyrosynthetic techniques, as did Dr. J. W. Earley.

Professor Peacock kindly obtained a type specimen of aramayoite from Harvard Museum, and one of miargyrite from the University of California collection.

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XANTHOPHYLLITE*

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ABSTRACT

New observations are made on two of the three known occurrences of xanthophyllite *var.* valuevite and compared with the published data on the third. These led to the following descriptions of the two. Valuevite from Achmatovsk, Russia is monoclinic with space group $C2/m$; the unit cell with $a=5.24$, $b=8.98$, $c=9.79$ kX, $\beta=100°10'$, $a:b:c=0.584:1:1.090$ contains $Ca_2(Mg_4Al_2)(Al_{5.6}Si_{2.4})O_{20}(OH)_4=2[Ca(Mg_2Al)(Al_{2.8}Si_{1.2})O_{10}(OH)_2]$; measured specific gravity $=3.076$. Optically negative, $\beta=1.662$, $\gamma=1.663$, $2V=19°30'$ (all Na light).

Valuevite from Crestmore, California is chemically and crystallographically identical to Achmatovsk valuevite, the unit cell with $a=5.204$, $b=8.995$, $c=9.833$ kX, $\beta=100°04'$, $a:b:c=0.5785:1:1.0987$, measured specific gravity 3.076. Optically negative, $\beta=1.659$, $\gamma=1.660$, $2V=21°15'$ (all Na light). An indexed powder pattern is given for Crestmore valuevite.

Some doubt is raised as to whether the original yellow-coloured xanthophyllite is the same species as the green variety.

The brittle mica xanthophyllite was first described by Rose in 1840 as a wax-yellow mineral occurring near Zlatoust in the southern Urals of Russia. In view of its poor crystallinity, a crystallographic study was not made, but its general physical properties as well as several chemical analyses were published. Three further occurrences are recorded in the literature. Koksharov (1875) described a green variety, occurring near Achmatovsk (15 kilometres north of Zlatoust), to which he gave the varietal name waluewite [valuevite], because of the differences in colour and optics. Eakle (1916) and Sanero (1940) described occurrences of the green variety from Crestmore, California and Adamello, Italy, respectively. The latter two were identical with valuevite in all comparable properties.

Although a thorough study of the Adamello valuevite was made by Sanero, *x*-ray diffraction work has not been performed on any of the previous occurrences. The present paper gives the results of a mineralogical study of valuevite from both Achmatovsk and Crestmore. Unfortunately a sample of the original xanthophyllite could not be obtained, and there is some doubt as to whether the yellow and green varieties are actually of the same species.

Material and acknowledgements. The samples consisted of several flakes of the poorly crystallised Crestmore material (ROMM 16622) and one crystal of the Achmatovsk material (ROMM 6362) which was sufficiently well crystallised to permit rough measurement of four forms on the optical goniometer.

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The author is indebted to Dr. V. B. Meen, Director of the Royal Ontario Museum of Geology and Mineralogy, who provided the samples used in this study, and Dr. L. G. Berry of Queen's University, Kingston for taking precession pictures of the Crestmore material.

Physical properties. Specific gravity determinations were made on both samples, using the Berman balance. The results are given in Table 1, together with those of other observers.

TABLE 1. XANTHOPHYLLITE: SPECIFIC GRAVITY

	Crestmore	Achmatovsk	Adamello	Zlatoust
S.A.F.	3.076	3.076	—	—
Sanero	—	—	3.075	—
Eakle	3.081	—	—	—
Koksharov	—	3.093	—	3.035–3.062
Rose	—	—	—	3.044

It can be readily seen that the specific gravity of the yellow variety from Zlatoust is of the same order as for the three green varieties and it cannot be distinguished from them on that basis.

X-ray crystallography. Laue diffraction patterns indicated a monoclinic symmetry and permitted selection of the proper axes for single crystal work. Rotation and Weissenberg photographs were taken about the *a* and *b* axes and precession photographs about the *b*-axis. The systematically missing spectra, namely

(hkl) present only with $h+k=2n$
$(h0l)$ present only with $h=2n$
$(0k0)$ present only with $k=2n$

lead to three possible space-groups $C_s^3\text{-}Cm$, $C_2^3\text{-}C2$, and $C_{2h}^3\text{-}C2/m$ with $C_{2h}^3\text{-}C2/m$ being the most probable.

As shown in Table 2, the cell dimensions obtained agree fairly well with those obtained by Sanero on the Adamello material.

TABLE 2. XANTHOPHYLLITE: LATTICE DIMENSIONS[1]

	Crestmore	Achmatovsk	Adamello	
a	5.204 kX	5.24 kX	5.20 kX	(5.21A)[2]
b	8.995	8.98	9.00	(9.02)
c	9.833	9.79	9.95	(9.97)
β	100°04′	100°10′	100°03′	

[1] Using CuKα_1=1.5374 kX

[2] Values given by Sanero, possibly they are in kX units.

Geometrical crystallography. Measurements were made on the one crystal from Achmatovsk on the optical goniometer. The reflections were very poor and since the only good reflecting face was the base *c*(001), the crystal was set up in a polar position and the angles between the base and three other observable forms were measured. The indices were determined from a stereographic projection. No effort was made to determine an axial ratio from the goniometric measurements. Table 3 shows the forms listed by Hintze (1897) who adopted Koksharov's setting, Dana (1892) who adopted Tschermak's setting, and the forms observed by the author. The indices of all the forms in the new setting are listed in column 1.

TABLE 3. XANTHOPHYLLITE: FORMS

New Setting	Hintze	Dana	S.A.F.
c(001)	*c*(001)	*c*(001)	(001)
x(301)	*x*(101)	*x*($\bar{1}$02)	(301)
z(905)	*Z*(304)	*z*($\bar{3}$08)	
t(011)	*t*(023)	*t*(013)	
v(023)	*v*(049)	*v*(029)	
r(034)	*r*(012)	*r*(014)	
Y(038)	*Y*(014)	*Y*(018)	
h(0.9.16)	*h*(038)	*h*(0.3.16)	
σ(335)	*s*(113)	*σ*($\bar{1}$16)	
o(337)	*o*(114)	*o*($\bar{1}$18)	
ω(338)	*ω*(229)	*ω*($\bar{1}$19)	(338)
k($\bar{1}$13)	*o′*($\bar{1}$14)	*k*(118)	
N($\bar{3}$31)	*N*(110)	—	
s($\bar{3}$37)	*s′*($\bar{1}$13)	*s*(116)	
w($\bar{3}$.3.10)	—	*w*(119)	
L($\bar{3}$91)	*L*(130)	*L*(130)	
d($\bar{3}$95)	*d*($\bar{1}$32)	*d*(134)	($\bar{3}$95)
n($\bar{3}$.9.25)	*n*($\bar{1}$.3.12)	*n*(1.3.24)	

The axial ratio adopted by Hintze was

$$a:b:c=0.57736:1:1.62214;\quad \beta=90°0'$$

The transformation to the new setting is as follows:

$$\text{Hintze to S.A.F.}=100/010/\tfrac{\bar{1}}{3}0\tfrac{2}{3}$$

The axial ratio adopted by Dana was

$$a:b:c=0.57735:1:3.24427;\quad \beta=90°0'$$

The transformation to the new setting is as follows:

$$\text{Dana to S.A.F.}=\bar{1}00/0\bar{1}0/\tfrac{1}{3}0\tfrac{1}{3}$$

The transformation takes this form since Dana adopted Tschermak's setting which was front to back. The axial ratios derived using the *x*-ray cell dimensions are:

Achmatovsk material—$a:b:c=0.584:1:1.090$; $\beta=100°10'$
Crestmore material—$a:b:c=0.5785:1:1.0987$; $\beta=100°04'$

Dana's setting is comparable to a single-layer mica, as described by Hendricks (1939), which has been forced into a 3-layer setting. Koksharov's is similar, with the 3-layer *c*-axis cut in two, producing effectively a $1\frac{1}{2}$-layer mica.

TABLE 4. ACTUAL MEASUREMENTS ON ACHMATOVSK XANTHOPHYLLITE

	S.A.F.	Hintze	Dana
$c\omega(001):(338)$	35°41′ (2)	$35°47\frac{1}{3}'$	35°47′
$cx(001):(301)$	70 32 (1)	$70\ 24\frac{1}{2}$	$70\ 24\frac{1}{2}$
$cd(001):(\bar{3}95)$	70 30 (2)	$70\ 24\frac{1}{2}$	$70\ 24\frac{1}{2}$

The figures in parentheses following the author's measurements denote the number of measurements made.

The actual measurements made on the crystal are shown in Table 4 and are compared to those values listed by Hintze and Dana. Table 5 is an angle table calculated using the axial ratio derived by *x*-ray measurements on the Achmatovsk material.

Composition and cell content. Analyses for each occurrence of valuevite are shown in Table 6. Analysis I by Eakle was made on Crestmore material, and this, combined with the cell dimensions and specific gravity determined by the author, served to determine the structural formula. Number III by Clarke and Schneider (1892) and IV by Nikolajev (selected from Hintze) on Achmatovsk material were treated in the same manner. Number II on Adamello material was calculated using Sanero's cell dimensions and specific gravity.
The structural formulae are as follows:

$$\text{I}\quad 2[\mathrm{Ca_{0.98}(Mg_{2.09}Fe''_{0.02}Fe'''_{0.15}Al'''_{0.70})(Al_{2.83}Si_{1.17})O_{9.92}(OH)_{2.10}}]$$

$$\text{II}\quad 2[\mathrm{(Ca_{1.00}Na_{0.12}K_{0.01})(Mg_{2.23}Fe''_{0.06}Fe'''_{0.04}Al'''_{0.72})(Al_{2.78}Si_{1.22})O_{10.16}(OH)_{1.92}}]$$

$$\text{III}\quad 2[\mathrm{Ca_{1.00}(Mg_{2.17}Fe''_{0.01}Fe'''_{0.12}Al'''_{0.68})(Al_{2.82}Si_{1.18})O_{9.90}(OH)_{2.15}}]$$

$$\text{IV}\quad 2[\mathrm{Ca_{0.98}(Mg_{2.14}Fe''_{0.04}Fe'''_{0.08}Al'''_{0.76})(Al_{2.84}Si_{1.16})O_{9.97}(OH)_{2.07}}]$$

It seems obvious that the chemical composition is constant from one oc-

TABLE 5. XANTHOPHYLLITE—$Ca_2Mg_4Al_2(Al_{5.6}Si_{2.4})O_{20}(OH)_4$

Monoclinic—$C2/m$; $a:b:c=0.584:1:1.090$; $\beta=100°10'$
$p_0:q_0:r_0=1.868:1.073:1$; $\mu=79°50'$
$p_0'=1.898$, $q_0'=1.090$, $x_0'=0.179$

Form	ϕ	ρ	ϕ_2	$\rho_2=B$	C	A
$c(001)$	90°00′	10°10′	79°50′	90°00′	0°00′	79°50′
$x(301)$	90 00	80 20½	9 40	90 00	70 10	9 40
$z(905)$	90 00	74 27½	15 32½	90 00	64 17½	15 32½
$t(011)$	9 20½	47 51	79 50	42 59	47 01	83 05½
$v(023)$	13 51½	36 49	79 50	54 25½	35 34½	81 45
$r(034)$	12 22	39 56	79 50	51 10½	38 49½	82 06
$Y(038)$	23 41	24 03½	79 50	68 05	21 55	80 34½
$h(0.9.16)$	16 18	32 34½	79 50	58 53	31 07	81 18½
$\sigma(335)$	63 36½	55 48	37 11	68 26	46 50½	42 11½
$o(337)$	64 48	47 39½	45 12½	71 39½	38 38½	48 02
$\omega(338)$	65 21½	44 26	48 17½	73 01½	35 16½	50 29
$k(\bar{1}13)$	−51 17½	30 09½	114 23½	71 41	38 33	113 05
$N(\bar{3}31)$	−59 20	81 00	169 43½	59 44	89 54½	140 11½
$s(\bar{3}37)$	−53 37½	38 13½	122 23	68 28	46 44½	119 53
$w(\bar{3}.3.10)$	−50 01	26 59	111 18½	73 03	35 20	110 20½
$L(\bar{3}91)$	−29 20½	84 55½	169 43½	29 44½	89 56½	119 13
$d(\bar{3}95)$	−26 03½	65 24	133 49	35 14	70 10½	113 32½
$n(\bar{3}.9.25)$	− 7 02½	21 34½	92 46½	68 35½	24 51½	92 35

TABLE 6. XANTHOPHYLLITE: CHEMICAL ANALYSES

	I	II	III	IV
SiO_2	16.74	17.11	16.85	16.39
TiO_2	—	tr.	tr.	—
Al_2O_3	42.70	41.71	42.33	43.40
Fe_2O_3	2.85	0.80	2.35	1.57
FeO	0.41	0.94	0.20	0.60
MnO	—	0.03	—	—
MgO	20.03	21.03	20.77	20.38
CaO	13.09	13.17	13.30	13.04
BaO	—	tr.	—	—
Na_2O	—	0.89	—	—
K_2O	—	0.12	—	—
H_2O	4.49	4.03	4.60	4.40
	100.31	99.83	100.40	99.78
S.G.	3.076(S.A.F.)	3.075(San)	3.076(S.A.F.)	3.076(S.A.F.)

currence to another. The ideal formula is hence

$$2[Ca(Mg_2Al)(Al_{2.8}Si_{1.2})O_{10}(OH)_2].$$

An extremely interesting feature is the Al:Si ratio which averages out to approximately Al:Si=2.80:1.20. This is a much greater substitution of aluminium for silicon than is normally observed in the platy silicate minerals. It is approaching a 3:1 ratio which may indeed be the ideal ratio for the brittle micas. The extra charge on the silicon-oxygen sheet probably accounts for the slightly shorter *c*-axis than that observed in the dark-coloured micas, and explains to some extent the brittle nature of this mica.

The published analyses for the yellow xanthophyllite are extremely similar to those tabulated above. No attempt was made to work out its cell content using the cell dimensions of the green variety since this would imply crystallographic identity which has definitely not been established.

Optical properties. Optical studies were carried out on the materials from both Achmatovsk and Crestmore using Na light. Table 7 shows the values obtained as well as those of Eakle and Sanero:

TABLE 7. XANTHOPHYLLITE: OPTICAL DATA, $Y=b$, OPTICALLY NEGATIVE

	α	β	γ	2V
Material Crestmore, S.A.F.	—	1.659	1.660	21°15′
Material Achmatovsk, S.A.F.	—	1.662	1.663	19°30′
Material Crestmore, Eakle	—	1.660	1.660	12°–18°
Material Adamello, Sanero	—	—	—	29°50′ (2E)

Rose describes xanthophyllite (yellow variety) as being effectively uniaxial.

X-ray powder pattern. Sufficient material was available to make an *x*-ray powder pattern of the Crestmore material only. Table 8 lists the observed lines (Cu radiation) with their measured and calculated spacings. The intensities are based on 10 for the strongest line. The pattern was indexed, in part, using the cell dimensions derived by the single crystal *x*-ray work.

Conclusions. A complete mineralogical description of the yellow variety of xanthophyllite including its morphology and *x*-ray crystallography was not made, since none of it is available for study. However, the chemical analyses of the yellow and green varieties are almost identical, but the optical properties as described by Rose (yellow and almost uniaxial) differ from those shown for valuevite. Nevertheless, there is insufficient

TABLE 8. XANTHOPHYLLITE: $Ca(Mg_2Al)(Al_{2.8}Si_{1.2})O_{10}(OH)_2$:
X-RAY POWDER PATTERN

Monoclinic, *C*2/*m*; $a=5.204$, $b=8.995$, $c=9.833$ kX; $\beta=100°04'$; $Z=2$

I	*d*(meas.)	(*hkl*)	*d*(calc.)	*I*	*d*(meas.)	(*hkl*)	*d*(calc.)
2	4.52 kX	(020)	4.50 kX	½	1.690	(240)	1.690
3	3.57	$(\bar{1}12)$	3.56			$(\bar{1}51)$	1.689
8	3.23	(003)	3.23	½	1.656	$(\bar{2}42)$	1.656
1	2.84	$(\bar{1}13)$	2.83			(151)	1.656
10	2.56	(200)	2.56	3	1.614	(006)	1.614
		(032)	2.55	7	1.499	(060)	1.499
		$(\bar{1}31)$	2.56	7	1.484	(330)	1.484
1	2.44	$(\bar{2}02)$	2.45	1	1.362	(225)	1.362
		(113)	2.44			(332)	1.362
		(131)	2.45			$(\bar{3}34)$	1.361
1	2.36	(201)	2.38	1	1.313		
		(014)	2.34	½	1.292		
		$(\bar{1}32)$	2.37	½	1.274		
1	2.20	$(\bar{2}03)$	2.20	½	1.242		
		(132)	2.20	½	1.185		
5	2.11	(202)	2.12	½	1.099		
		(221)	2.10	½	1.055		
		$(\bar{1}33)$	2.11	½	1.019		
3	1.936	(005)	1.936	½	0.981		
		$(\bar{2}04)$	1.937	½	0.961		
		(133)	1.937	½	0.932		
½	1.850	$(\bar{1}34)$	1.851	½	0.886		
½	1.780	(025)	1.779	½	0.862		
		$(\bar{2}24)$	1.779	½	0.809		
				½	0.787		

evidence to suggest that xanthophyllite and valuevite are not varieties of the same species of brittle mica.

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EMPRESSITE AND "STUETZITE"

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Abstract

Empressite, from Empress Josephine mine, Colorado (type specimen), stuetzite, the original crystal described by Schrauf (1878), a homogeneous fusion with composition Ag_5Te_3 and hydrosynthetic crystals (Ag-Te) give identical *x*-ray powder patterns.

The crystal of stuetzite and the synthetic crystals are hexagonal with probably space group $C6/mmm$, $a=13.46$ $c=8.46$ kX. The *x*-ray powder pattern is readily indexed with these dimensions. Analyses indicate the composition AgTe or Ag_7Te_5 for natural empressite. Fusions of the elements clearly indicate a structural formula 7 $[Ag_5Te_3]$, close to $3[Ag_{12}Te_7]$ (Koern, 1939). A possible general structural formula would be $18[Ag_{2-x}Te_{1+x}]$ where x may have values 0.1 to 0.5 with corresponding calculated specific gravities 7.73 to 7.90 in fair agreement with measured values 7.99 (Ag_5Te_3 fusion) and 7.61 (empressite).

This joint work is the result of studies which commenced in 1944 when Thompson (1946) made a broad study of the descriptive mineralogy and occurrence of telluride minerals. A short description of empressite with *x*-ray powder data was published by Thompson (1949). Further studies were made possible by the availability of the original crystal of stuetzite kindly loaned by Professor Machatschki from the Vienna Museum. In May and June of 1950, Professor Peacock remeasured the stuetzite crystal and also obtained single crystal *x*-ray measurements on it. This was the last laboratory work done by Professor Peacock. During 1948–49 in the course of a study of hydrosynthesis of silver and gold tellurides, Rowland (1950) obtained crystals which gave an *x*-ray powder pattern identical with the pattern of empressite. The present account, prepared by Berry with the help of Thompson, has been largely extracted from the unpublished theses of Thompson (1946) and Rowland (1950) and from Professor Peacock's notes.

Previous Observations

Empressite, a new silver telluride, from the Empress Josephine Mine, Kerber Creek District, Colorado, was discovered and named by Professor R. D. George of the University of Colorado, and later described by Bradley (1914, p. 63) with two analyses in good agreement with the formula AgTe. The mineral was described as massive to finely granular with no indication of crystal form; brittle to friable with a finely conchoidal to uneven fracture. Hardness between 3 and $3\frac{1}{2}$; specific gravity 7.510; lustre metallic and colour pale bronze.

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The blowpipe reactions as given by Bradley are: in the oxidizing flame on charcoal it fuses at $F=1$ giving a heavy white coating of tellurium dioxide and a black globule, which if placed in the reducing flame gives on cooling white dendritic points of silver on its surface. Prolonged heating in the oxidizing flame gives a globule of silver. In the open tube a faint white sublimate of tellurium dioxide is formed which if strongly ignited fuses to colourless globules. The mineral is readily soluble in hot dilute nitric acid.

The mineral is not described by Short (1940), but Mathewson (in Bradley, 1914) stated that empressite is structurally homogeneous with large irregular light and dark polygonal grains and no interstitial matter. There is no published x-ray information on the mineral.

Stuetzite, was described by Schrauf (1878) in highly modified hexagonal or pseudohexagonal crystals from a single specimen in the mineral collections of Vienna University; the locality was probably Nagyág, Transylvania. It is associated with gold and hessite on quartz. The composition was given as Ag_4Te; the silver was determined approximately with the blowpipe.

The mineral was described as lead-grey with reddish tinge, lustre metallic, fracture uneven to subconchoidal. It is easily fusible to a dark bead from which a silver globule is obtained by reduction with soda and yields tellurium dioxide in the open tube. Stuetzite is not described by Short (1940).

MATERIAL STUDIED

1. Empressite, Empress Josephine Mine, Kerber Creek District, Colorado (USNM, R 7243, type specimen). A compact mass of empressite partially covered with a coating of a clay-like material. On one corner is a small area of galena showing cubic cleavage. Small amounts of yellow-brown sphalerite and finely crystalline pyrite are disseminated throughout the clay-like material.

2. Empressite, Red Cloud Mine, Boulder County, Colorado (mis-labelled petzite). Empressite and altaite sparsely disseminated throughout rock.

3. Stuetzite, probably from Nagyág, Transylvania (Mineralogical Museum of Vienna University, 5808). Original crystal described by Schrauf (1878),

4. "Empressite," probably part of original material collected by Dr. George from Empress Josephine mine, Colorado. Obtained from University of Colorado collections through E. M. Gunnell, Denver.

PHYSICAL AND MICROSCOPICAL OBSERVATIONS

In materials 1 and 2 empressite has a heavy metallic lustre and an almost jet black colour like petzite but with a slight bronzy cast. It has no cleavage, and is quite brittle, with a finely conchoidal to uneven fracture.

The hardness of $3\frac{1}{2}$ and the specific gravity measured on several fragments is 7.61 ± 0.01, as compared to 7.51 (Bradley). Empressite polishes to a smooth surface which shows intense reflection pleochrism, light gray-mauve to creamy-white, depending on the orientation (Fig. 1). The anisotropism is likewise very strong with polarization colours white, yellowish-green, russet-brown, brown, dark blue. Twinning and evidence of cleavage are absent. The mineral makes a solid moderate coarse grained mosaic (Fig. 2).

Etch-reactions: HNO_3 slow effervescence, slowly stains iridescent which may be removed by hard buffing; HCl negative; KCN negative; $FeCl_3$ stains iridescent and remains on buffing; KOH negative; $HgCl_2$ quickly stains iridescent and remains on buffing. The hardness estimated with a needle is C.

The sections examined consisted essentially of empressite with small amounts of altaite and pyrite which are present as inclusions in the empressite (Fig. 1). These inclusions were identified by an x-ray powder photograph on material 1. Altaite forms ill-defined stringers with small blebs of chalcopyrite and sphalerite which cut the empressite in the type material. A few small crystals of pyrite and marcasite are completely surrounded and corroded by the empressite. Galena, noted on the hand specimen but not observed in polished section, was checked with an x-ray powder photograph on material 1. It gave a positive test for Te with sulphuric acid.

An analysis of 250 mg. of the type material, carefully selected under the binocular microscope was made by R. N. Williams with the following result: Ag 54.77, Te 45.26, total 100.03. This differs substantially from the previous analyses and corresponds to Ag_4Te_3 (Ag 53.0, Te 47.0) or Ag_7Te_5 (Ag 54.2, Te 45.8). In reporting these results the analyst wrote: "The impurities which consist of gold, lead, iron and copper, do not in my opinion exceed 0.10 per cent." The new analysis indicates distinctly more silver than that required by the original formula AgTe but not as much as is required by the composition Ag_5Te_3 suggested by the fusion experiments described later.

Polished section observations could not be made on the crystal of stuetzite. A small fragment of the crystal, which became detached in transit, yielded the same x-ray powder pattern as materials 1 and 2.

Material 4, consisting of a few fragments, showed physical properties similar to the other empressite specimens. The specific gravity measured on several pieces is $7.30 \pm .04$. In polished section the mineral is similar to empressite and associated with altaite. A spectrographic analysis showed mainly silver and tellurium with minor lead and traces of iron, copper and silicon. This material gave an x-ray powder pattern (table 5)

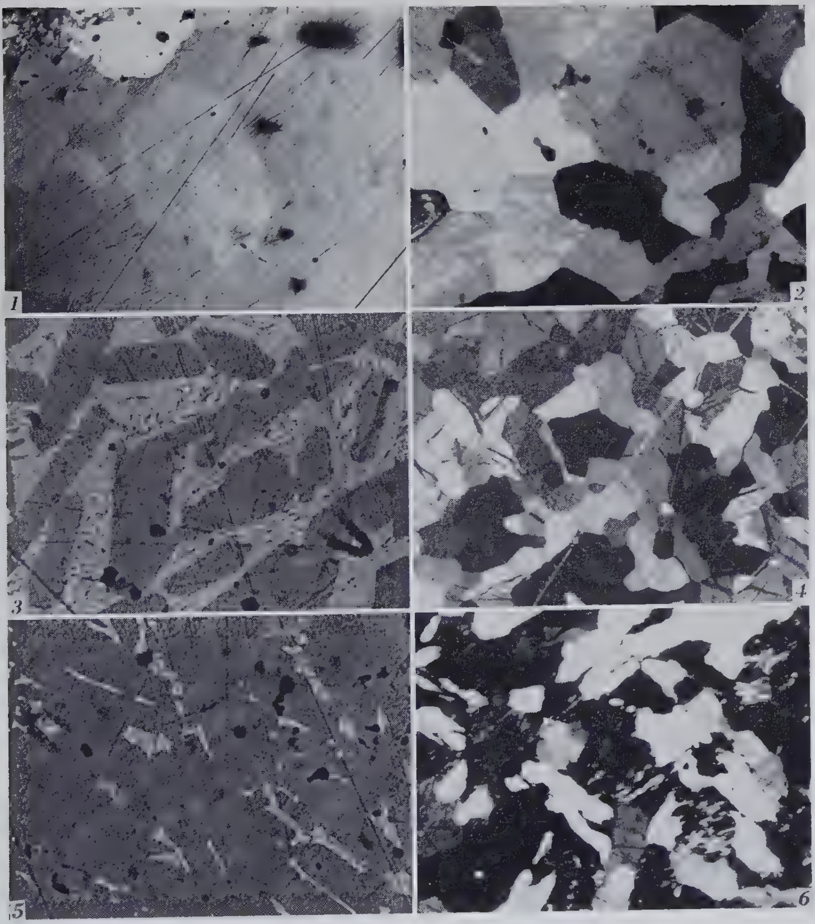

Fig. 1, 2.—Empressite, Empress Josephine mine, Kerber Creek District, Colorado (material 1). Polished section; ×135. Fig. 1. Showing intense reflection pleochroism with a white inclusion of altaite (one nicol). Fig. 2. Coarse mosaic showing strong anisotropism (crossed nicols).

Figs. 3–5.—Ag-Te fusion products in polished section. Fig. 3. Ag:Te=1:1, laths of empressite (gray) in an eutectic groundmass of empressite and tellurium (one nicol, ×290). Fig. 5. Ag:Te=3:2, empressite (gray) with elongate streaks of tellurium (white) (one nicol, ×290). Fig. 4. Ag:Te=5:3, homogeneous mosaic with strong anisotropism identical with empressite (crossed nicols, ×135). Fig. 6. Ag:Te=7:4, interlocking crystals of empressite (black and white) with interstitial patches of twinned hessite (crossed nicols, ×290).

unlike empressite or any other known telluride minerals. A few small fragments were fused in vacuum; the fusion product gave the powder pattern of empressite plus the strong lines of clausthalite.

SYNTHESIS OF EMPRESSITE

Pyrosyntheses.

Charges with the following compositions were prepared in an endeavour to synthesize empressite:

TABLE 1. Ag-Te FUSIONS

	Composition			Products	S.G. (meas.)
	Atomic Prop.	Ag %	Te %		
1	1 Ag: 1 Te	45.8	54.2	empressite* tellurium*	—
2	7 Ag: 5 Te (near)	54.77	45.26	empressite* hessite tellurium	—
3	3 Ag: 2 Te	55.9	44.1	empressite* tellurium*	—
4	5 Ag: 3 Te	58.5	41.5	empressite* homogeneous	7.99
5	12 Ag: 7 Te	59.2	40.8	empressite* hessite*	8.04±0.03
6	7 Ag: 4 Te	59.6	40.4	empressite* hessite*	8.02±0.01

* Identified by *x*-ray powder patterns.

1. Composition from original analysis of empressite. 2. Composition from analysis by Williams. 3. Composition preferred by Hansen (1936). 4. Gives homogeneous product. 5. Composition given by Koern (1939). 6. Composition given by Chikashigi & Saito (1916).

The charge Ag:Te=1:1 fused fairly readily and produced on cooling a stony, black, brittle regulus. A polished section showed a non-homogeneous product consisting of two phases: large laths of a gray phase with polarization colours like those of empressite, in a groundmass consisting of an eutectic between the gray phase and a white phase resembling tellurium (Fig. 3). An *x*-ray powder photograph identified these two phases as empressite and tellurium.

The charge with the composition corresponding to Williams' analysis, Ag 54.77, Te 45.26, fused fairly readily with slight green fluorescence to a black, splendent, brittle, steel-gray, somewhat pitted regulus but with indication of crystallinity on the lower side. A polished section showed an inhomogeneous product consisting of an intimate intergrowth of three phases. The main component occurs in lath-like gray crystals with polarization colours similar to those of empressite. A creamy-gray phase with low polarization colours similar to those of hessite contains pinkish-white streaks of a third phase which resembles native tellurium. In places there is a delicate eutectic between the gray and creamy-gray phases. An x-ray powder photograph of a random fragment gave only the empressite pattern.

The charge $Ag:Te=3:2$ fused at a fairly high temperature and produced on cooling a black, stony, brittle regulus which developed a sprout and several small spherical globules on its surface. A polished section showed an inhomogeneous product which consisted of two phases. The major phase, a gray-white substance encloses many elongated stringers of a pinkish white phase (Fig. 5). Under crossed nicols the major phase shows irregular lath-like crystals. An x-ray powder photograph of a fragment from the main regulus gave the empressite pattern. An x-ray powder photograph of the main sprout on the regulus gave the empressite pattern plus a few lines of tellurium.

The charge $Ag:Te=5:3$ fused readily with slight green fluorescence and produced a cooling in gray, brittle regulus. On a fresh fracture the colour is lustrous black, the most "empressite looking" of all the fusions. This regulus as well as all previous ones shows no cleavage. It has however, unlike the previous fusion products, a finely conchoidal fracture similar to that of empressite. A polished section revealed a homogeneous product, gray-white in colour with very weak pleochroism. With crossed nicols it makes a moderate, approximately equidimensional mosaic with polarization colours like those of empressite (Fig. 4). An x-ray powder photograph gave only the empressite pattern.

The charge $Ag:Te=12:7$ fused readily and produced a regulus with several small beads showing poorly developed faces, on its upper sooty-black surface. The lower half of the regulus is porous with a shiny black colour. A polished section shows an inhomogeneous product consisting of a major gray phase (empressite) and a lesser amount of a bluish-white phase (hessite) as irregular areas.

The charge $Ag:Te=7:4$ fused fairly readily and produced a lustrous, black, stony, brittle regulus. A polished section showed an intimate intergrowth of two phases which were identified by an x-ray powder photograph as empressite and hessite (Ag_2Te). The empressite occurs in elongated grains with small interstitial areas of hessite which show confused lamellar twinning (Fig. 6).

On heating several fragments of empressite in an evacuated silica glass tube, a black vapour, presumably tellurium was observed creeping up the sides of the tube. The black somewhat sectile regulus was too small for a polished section study but an *x*-ray powder photograph of a fragment gave the hessite (Ag_2Te) pattern. Apparently the empressite lost some of its tellurium and formed hessite.

The results of this brief study of the silver-tellurium system are not in full accord with the results of previous workers (Hansen, 1936, p. 65) who reported two compounds: one with the composition Ag_2Te corresponding to the mineral hessite, which was produced synthetically and the other with a doubtful composition, Ag_3Te_2, or AgTe, presumably corresponding to empressite. Our results indicate a homogeneous product similar to empressite with the composition Ag_5Te_3 while Chikashige & Saito (1916, p. 368) state that Ag_2Te reacts with the melt at 443° and forms another compound Ag_7Te_4 which changes at 403° to the β form. Koern (1939) reports, in abstract, finding two compounds Ag_2Te and $Ag_{12}Te_7$ both in two modifications.

Hydrosynthesis. In the course of study of the hydrosynthesis of gold and silver tellurides (Rowland, 1950), artificial crystals of hessite, calaverite and empressite were formed. The synthesis were performed in the type of graphite lined steel bomb developed by Dr. F. G. Smith at the University of Toronto and used in synthesis of ore minerals by Béland (1948), Robinson (1948) and others. Empressite was recognized in the products of two bomb runs designated VI & VII; the composition of the charges and the conditions are shown in table 2.

Table 2. Empressite: Composition of Bomb Charges

No.	$Na_2S\cdot 9H_2O$	Ag	Au	Te	H_2O	Time	Temp.	Products
VI	10 g.	0.5 g.	—	0.6 g.	20 cc.	4 days	250° C.	empressite
VII	20 g.	0.27 g.	0.49 g.	0.62 g.	20 cc.	25 hrs.	280° C.	empressite calaverite

The crystals of empressite produced in run VI were blocky, hexagonal and commonly in crystalline aggregates displaying parallel growth and purple tarnish. Those produced by run VII were untarnished silvery and blocky, usually as single crystals with well defined prismatic zone and poor terminal faces. The crystals were rather poorly formed and from 0.1 to 0.25 mm. in greatest dimension.

CRYSTALLOGRAPHY

The *x*-ray powder pattern of empressite was established on type material (1) by Thompson (1946, 1949). The same pattern was also given by material 2 and a homogeneous fusion of composition Ag_5Te_3. The pattern was relatively complex and no attempt was made to index it. In 1949, Rowland (1950) obtained synthetic empressite crystals giving an x-ray powder pattern identical with the pattern of natural empressite.

Synthetic crystals. Fifteen crystals of empressite from two bomb runs (VI and VII) were measured on the two-circle optical goniometer and the measured ϕ and ρ angles plotted in gnomonic projection. The projections clearly indicate hexagonal symmetry and normal indexing of the projections leads to the axial ratio from the ϕ and ρ measurements.

$$a:c=1:0.6323$$

The forms observed on nine measured crystals are listed in Table 3 with measured and calculated ϕ and ρ angles.

A single crystal, adjusted to rotate about the c-axis on the optical goniometer, yielded sharp rotation, zero and first layer Weissenberg films. The reciprocal lattice projections clearly indicate a hexagonal lattice with unit cell dimensions:

$$a=13.46, \quad c=8.45 \text{ kX};^1 \qquad a:c=1:0.628$$

The observed diffractions ($000l$ not observed) showing no systematic extinctions, are characteristic of the space group $C6mmm$ of the crystals belong to the holohedral class.

Original crystal of stuetzite. This crystal (about $\frac{3}{4}$ mm across), first measured by Schrauf (1878), was remeasured by Peacock. In his original notes he recorded measurements for 27 faces on one end and 28 faces on the other end (Fig. 7). He noted that "the projection of the measured crystal suggests Schrauf's $(10\bar{1}2)$ as the better unit form" and deduced, from the measurements, the hexagonal axial ratio in close agreement with Schrauf's value with c divided by two.

$$a:c \quad =1:0.6287 \text{ (M.A.P.)}$$
$$a:c/2=1:0.6265 \text{ (Schrauf, 1878)}$$

Peacock also noted that there seems to be no reason for not placing the mineral in the hexagonal system. Schrauf had suggested that it was pseudohexagonal, probably monoclinic.

In table 3 the measured ϕ and ρ angles for artificial empressite crystals and the original stuetzite crystal are given with angles calculated from the axial ratio. In Schrauf's setting all l indices are doubled.

Peacock obtained rotation and zero layer Weissenberg films from the stuetzite crystal. The rotation film about c is poor due to the large size of the crystal and did not yield sharp measurements. The zero layer Weis-

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[1] Using wavelength CuKα 1.5374.

TABLE 3. EMPRESSITE: MEASURED AND CALCULATED TWO CIRCLE ANGLES

Hexagonal C6/mmm $a:c=1:0.6287$; $p_0:q_0=0.7260:1$

Form	Stuetzite (M.A.P.)			Artificial (J.F.R.) 9 crystals			Calculated	
	ϕ	ρ	obs. faces	ϕ	ρ	obs. forms	ϕ	ρ
$c(0001)$	—	0°00′	1	—	0°00′	8	—	0°00′
$m(10\bar{1}0)$	29°53′	90 00	2	30°05′	90 00	9	30°00′	90 00
$a(11\bar{2}0)$	−0 12	90 00	2	0 15	90 00	9	00 00	90 00
$(32\bar{5}0)$	7 34	90 00	1	—	—	—	6 35	90 00
$(53\bar{8}0)$	—	—	—	8 05	90 00	2	8 13	90 00
$h(21\bar{3}0)$	10 52	90 00	2	10 20	90 00	3	10 54	90 00
$l(31\bar{4}0)$	16 10	90 00	2	16 45	90 00	3	16 06	90 00
$(41\bar{5}0)$	—	—	—	19 35	90 00	1	19 06	90 00
$d(10\bar{1}2)$	29 58	20 15	2	29 50	20 21	3	30 00	19 57
$(20\bar{2}3)$	—	—	—	29 45	26 08	3	30 00	25 49½
$f(10\bar{1}1)$	29 59	36 08	3	29 40	36 14	6	30 00	35 58½
$(40\bar{4}3)$	—	—	—	30 15	45 20	1	30 00	44 04
$(30\bar{3}2)$	30 09	47 19	1	—	—	—	30 00	47 26
$g(20\bar{2}1)$	30 00	55 18	3	30 25	55 20	5	30 00	55 26½
$s(30\bar{3}1)$	30 01	65 15	5	29 30	63 15	1	30 00	65 20
$u(11\bar{2}3)$	0 24	23 03	3	0 40	22 42	2	0 00	22 44½
$z(11\bar{2}2)$	0 08	32 22	4	0 25	32 21	5	0 00	32 09½
$(22\bar{4}3)$	0 04	40 00	1	0 05	40 40	1	0 00	39 58½
$(33\bar{6}4)$	—	—	—	0 15	43 25	2	0 00	43 19
$y(11\bar{2}1)$	0 04	51 22	4	0 05	51 47	6	0 00	51 30½
$x(22\bar{4}1)$	0 04	68 09	3	0 00	68 15	1	0 00	68 19
$(53\bar{8}2)$	7 36	68 02	1	—	—	—	8 13	68 31
$(21\bar{3}2)$	10 37	43 35	1	—	—	—	10 54	43 50½
$i(21\bar{3}1)$	10 48	63 23	6	—	—	—	10 54	62 30
$(31\bar{4}3)$	17 00	41 14	1	—	—	—	16 06	41 06½
$o(31\bar{4}1)$	16 00	68 53	2	—	—	—	16 06	69 05½
$(41\bar{5}1)$	18 12	72 19	1	—	—	—	19 06	73 16

senberg shows distinct hexagonal symmetry and the two films lead to the lattice dimensions:

$$a=13.4, \qquad c=8.5\pm0.1\ \text{kX}.$$

Koern (1939) gives hexagonal lattice dimensions for α-$Ag_{12}Te_7$ which compare closely with our measurements for empressite and stuetzite.

$a=13.429$	$c=8.4508$ kX	Koern (1939)
$a=13.4$	$c=8.5$ kX	Stuetzite (M.A.P.)
$a=13.46$	$c=8.45$ kX	Artificial crystals (J.F.R.)
$a=13.46$	$c=8.46$ kX	Empressite (powder data)

X-ray powder pattern. Empressite, materials 1 and 2, stuetzite, the homogeneous fusion Ag_5Te_3 and the hydrosynthetic crystals of Ag-Te all give an identical *x*-ray powder pattern. The observed intensities, measured θ values and interplanar spacings for type empressite from the Empress Josephine Mine, Kerber Creek District, Colorado (Thompson, 1949) are given in Table 4 together with the indices and calculated spacings for all the observed lines. There is a good agreement between each measured spacing and one or more spacings calculated from the lattice dimensions.

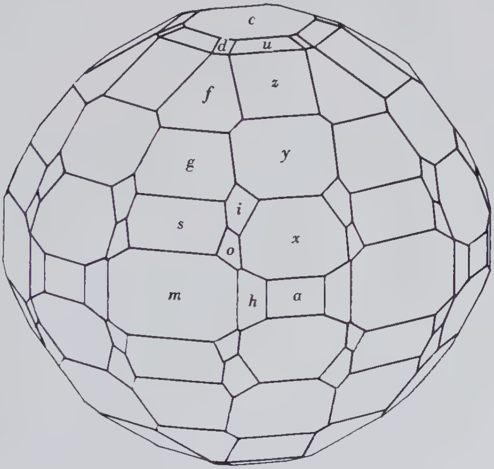

FIG. 7.—Stuetzite, original crystal described by Schrauf (1878), idealized drawing (L. G. B.) from sketches by M. A. Peacock. The crystal actually shows two $(h0\bar{h}l)$ and two $(h.h.\overline{2h}.l)$ zones complete from (0001) to $(000\bar{1})$, the forms shown are *c*(0001), *m*$(10\bar{1}0)$, *a*$(11\bar{2}0)$, *h*$(21\bar{3}0)$, *d*$(10\bar{1}2)$, *f*$(10\bar{1}1)$, *g*$(20\bar{2}1)$, *s*$(30\bar{3}1)$, *u*$(11\bar{2}3)$, *z*$(11\bar{2}2)$, *y*$(11\bar{2}1)$, *x*$(22\bar{4}1)$, *i*$(21\bar{3}1)$, *o*$(31\bar{4}1)$.

Empressite? (material 4) said to be part of the original material collected from the Empress Josephine mine gave a different *x*-ray powder pattern. The observed intensities, measured θ values and interplanar spacing from this pattern are given in Table 5. There is little hope of determining lattice dimensions from the material at present available.

Could this material really represent the original empressite with composition AgTe indicated by the early analyses? Or might it be the natural counterpart of the second modification of $Ag_{12}Te_7$ mentioned by Koern (1939) without any descriptive details? The pattern is recorded here in the hope that it might be recognized elsewhere.

TABLE 4. EMPRESSITE-$Ag_{2-x}Te$: *X*-RAY POWDER PATTERN (R.M.T.)

Hexagonal, *C*6/*mmm*; $a=13.46$, $c=8.46$ kX; $Z=21$

I	θ(Cu)	*d*(meas.)	*hkīl*	*d*(calc.)
½	10.1°	4.38kX	$12\bar{3}0$	4.406kX
½	11.2	3.96	$01\bar{1}2$	3.976
			$12\bar{3}1$	3.908
1	12.5	3.55	$11\bar{2}2$	3.581
			$03\bar{3}1$	3.531
½	13.1	3.39	$02\bar{2}2$	3.424
			$22\bar{4}0$	3.365
½	13.8	3.22	$13\bar{4}0$	3.233
2	14.7	3.03	$12\bar{3}2$	3.051
			$13\bar{4}1$	3.020
½	15.8	2.82	0003	2.820
1	17.0	2.63	$22\bar{4}2$	2.633
5	17.6	2.54	$23\bar{5}1$	2.550
			$14\bar{5}0$	2.544
			$02\bar{2}3$	2.539
1	20.1	2.24	$05\bar{5}1$	2.248
			$33\bar{6}0$	2.243
10	20.8	2.16	$14\bar{5}2$	2.180
			$33\bar{6}1$	2.168
			$22\bar{4}3$	2.161
2	21.3	2.12	$24\bar{6}1$	2.132
			$13\bar{4}3$	2.125
			0004	2.115
1	22.2	2.03	$05\bar{5}2$	2.042
			$15\bar{6}1$	2.032
			$04\bar{4}3$	2.027
			$11\bar{2}4$	2.018
1	23.5	1.928	$06\bar{6}0$	1.943
			$23\bar{5}3$	1.940
			$34\bar{7}0$	1.916
½	23.8	1.905	$12\bar{3}4$	1.907
½	24.3	1.868	$34\bar{7}1$	1.869
			$25\bar{7}0$	1.867
½	27.6°	1.659kX	$07\bar{7}0$	1.665kX
			$35\bar{8}0$	1.665
			$23\bar{5}4$	1.659
			$44\bar{8}1$	1.650
½	29.9	1.542	$07\bar{7}2$	1.549
			$35\bar{8}2$	1.549
			$17\bar{8}0$	1.544
			$33\bar{6}4$	1.539
½	31.8	1.459	$04\bar{4}5$	1.463
			$08\bar{8}0$	1.457
½	32.25	1.441	$36\bar{9}1$	1.447
			$44\bar{8}3$	1.445
			$08\bar{8}1$	1.436
½	32.65	1.425	$06\bar{6}4$	1.431
			$23\bar{5}5$	1.430
			$27\bar{9}0$	1.424
			$34\bar{7}4$	1.420
2	34.8	1.347	$17\bar{8}3$	1.354
			$33\bar{6}5$	1.351
			$27\bar{9}2$	1.350
			$18\bar{9}1$	1.347
			$5.5.\overline{10}.0$	1.346
			$12\bar{3}6$	1.343
			$24\bar{6}5$	1.342
½	35.8	1.314	$45\bar{9}3$	1.319
			$44\bar{8}4$	1.317
			$15\bar{6}5$	1.316
			$3.7.\overline{10}.0$	1.311
½	36.6	1.289	$09\bar{9}0$	1.295
			$08\bar{8}3$	1.294
			$13\bar{4}6$	1.293
			$26\bar{8}4$	1.284
½	37.0	1.277	$09\bar{9}1$	1.280
			$06\bar{6}5$	1.275

COMPOSITION AND CELL CONTENT

The reported composition of the materials we find to be structurally identical are shown in Table 6 together with the measured specific gravity.

The volume of the unit cell determined for empressite is 1327.7 kX^3. Combined with the measured specific gravity, 7.61 we get M=6122. M

TABLE 5. EMPRESSITE?—(MATERIAL 4) X-RAY POWDER PATTERN (R.M.T.)

I	θ(Cu)	d(meas.)	I	θ(Cu)	d(meas.)	I	θ(Cu)	d(meas.)
1	11.15°	3.98 kX	2	22.2°	2.03 kX	½	35.35°	1.329 kX
2	11.8	3.76	2	22.6	2.00	1	36.05	1.306
3	13.45	3.30	½	23.15	1.955	½	36.55	1.291
2	14.1	3.16	2	23.75	1.909	1	42.85	1.130
2	15.65	2.85	1	26.0	1.754	1	43.95	1.108
10	16.65	2.68	½	27.75	1.651	½	48.45	1.027
½	17.3	2.59	2	29.45	1.563	1	50.15	1.001
½	17.95	2.49	½	30.6	1.510	1	52.25	0.972
1	18.55	2.42	4	31.15	1.486	2	54.15	0.948
4	19.5	2.30	1	32.5	1.431	1	56.15	0.926
7	20.35	2.21	1	33.45	1.395	½	57.25	0.914
½	20.7	2.17	4	34.55	1.355	2	58.35	0.903
4	21.25	2.12						

values and calculated specific gravities for some of the above compositions are given below.

26[AgTe]	M=6123.0	G(calc.) 7.61
4[Ag_7Te_5]	M=5374.0	G(calc.) 6.68
5[Ag_7Te_5]	M=6967.5	G(calc.) 8.66
7[Ag_5Te_3]	M=6455.6	G(calc.) 8.03
3[$Ag_{12}Te_7$]	M=6563.5	G(calc.) 8.16

The early analyses of empressite (Bradley 1914, 1915) and the measured specific gravity clearly lead to a unit cell content of 26[AgTe] with good agreement between measured and calculated specific gravities. Our observations on pyrosyntheses indicate a cell content of 7 [Ag_5Te_3] for which the measured and calculated specific gravities are in substantial agreement. This cell content is very similar to 3[$Ag_{12}Te_7$] found by Koern (1939), which requires a higher specific gravity. If the composition

TABLE 6. EMPRESSITE: COMPOSITION

	Ag	Te	Formula	S.G.	
Empressite	45.17	54.75	Ag Te	7.51	Bradley (1914)
(type locality)	43.70	53.84	Ag Te	—	Dittus, in Bradley (1915)
(type locality)	54.77	45.26	Ag_7Te_5	—	R. N. Williams
(type locality)	—	—	—	7.61	R.M.T.
Homogeneous fusion	58.5	41.5	Ag_5Te_3	7.99	R.M.T.
Artificial	59.2	40.8	$Ag_{12}Te_7$	—	Koern (1939)
Stuetzite			Ag_4Te	—	Schrauf (1878)

were written $Ag_{2-x}Te$ the artificial materials would have a cell content of $21[Ag_{2-x}Te]$ where x is about 0.3 but for the natural materials with $x=1$ or $\frac{3}{5}$, $Z=21$ would be much too low. Or, the formula might be expressed as $Ag_{2-x}Te_{1+x}$ and the cell contents indicated by the observed compositions would then be:

AgTe	$18[Ag_{2-0.5}Te_{1+0.5}]$	$M=6358.5$	G(Calc.) = 7.90
Ag_7Te_5	$18[Ag_{2-0.25}Te_{1+0.25}]$	$M=6259.4$	G(Calc.) = 7.78
Ag_5Te_3	$18[Ag_{2-0.12}Te_{1+0.12}]$	$M=6223.1$	G(Calc.) = 7.75
$Ag_{12}Te_7$	$18[Ag_{2-0.1}Te_{1+0.1}]$	$M=6216.1$	G(Calc.) = 7.73

This structural formula, $18[Ag_{2-x}Te_{1+x}]$, appears to reconcile the differences in composition between natural and artificial materials except that the calculated specific gravity is lower than the measured value for the artificial fusion material with higher silver content. A complete structure determination must be undertaken however before this formula can be accepted for empressite.

Since the composition of this mineral, although still in some doubt, is certainly close to that originally ascribed to empressite, and the composition given for stuetzite is quite different and based on very uncertain chemical methods, it is proposed that the name empressite be retained and the name stuetzite be dropped.

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THE STRUCTURAL LATTICE OF HESSITE[1]

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Abstract

Artificial crystals of hessite, formed from aqueous alkaline sulphide solutions at moderate temperatures, are of three types:

Type I. Orthorhombic, with probable space group *Immm*. The unit cell, with $a=16.27$, $b=26.68$, $c=7.55$A; $a:b:c:=0.610:1:0.283$ contains 48 [Ag_2Te]. Specific gravity 8.35 calculated, 8.41 measured on natural hessite (Thompson 1949). The crystals are small (0.2 to 0.3 mm.) and show the forms: *b*(010) *a*(100) *m*(110) *n*(120) *l*(130) *k*(150) *d*(011) *s*(132) *o*(121) *r*(141) (161) *p*(211) *q*(231) with axial ratio from two circle measurements: $a:b:c=0.599:1:0.284$.

Type II. Orthorhombic crystals showing the forms *c*(001) *a*(100) *l*(120) *m*(110) *n*(320) *d*(011) *p*(111) with axial ratio from two circle measurements: $a:b:c=0.776:1:0.650$.

Type III. Rough cubic crystals modified by the forms *o*(111) and *d*(011).

Type II and the cubic crystals do not give single crystal *x*-ray photographs but give the same *x*-ray powder photograph as Type I crystals and natural hessite. At 155° C. Ag_2Te is isometric with $a=6.64$ A and cell content 4[Ag_2Te].

Hessite occurs in hydrothermal veins with other tellurides and gold. In Canada it has been recognized in polished sections and by *x*-ray powder photographs, usually as small anhedral grains or compact masses, from numerous localities (Thompson 1949). Hessite has been described in highly modified, distorted cubic or pseudocubic crystals from Botés, Roumania and Boulder County, Colorado. In polished section hessite shows twinning lamellae which disappear at 149.5° C., the mineral also becomes isotropic above this temperature (Dana, 1944). On the basis of the morphology the crystals have been described as triclinic, orthorhombic, rhombohedral or monoclinic by different authors. Ramsdell (1925) showed that the *x*-ray pattern of hessite is not isometric. Rahlfs (1936) obtained an isometric powder pattern for artificial Ag_2Te at elevated temperature.

Tokody (1932, 1934) obtained monoclinic crystal lattice dimensions from powder photographs and one rotation photograph about the cube edge of a relic isometric form. Thompson (1949) reports that the *x*-ray powder pattern will not index with the elements given by Tokody. Koern

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[1] Extracted from an unpublished M.Sc. thesis by J. F. Rowland: An *x*-ray study of gold and silver tellurides—*Queen's University* 1950. This work was carried out with the aid of a bursary from the National Research Council (Canada) 1948–49, and is published with the permission of the Council, and with permission of the Director-General of Scientific Services, Department of Mines and Technical Services, Ottawa, Canada.

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(1939) reports in abstract that Ag_2Te can be formed in two modifications and gives orthorhombic crystal lattice dimensions for the lower temperature form which show some similarity to the monoclinic elements of Tokody.

SYNTHESIS OF HESSITE

Direct fusion of silver and tellurium, in vacuo, in the proportions 2:1 results in homogeneous material (Thompson, 1946, 1948), which gives the same *x*-ray powder pattern as natural hessite.

During a study of the synthesis of gold and silver tellurides from aqueous solutions (Rowland, 1950), crystals of hessite were obtained. The *x*-ray powder photograph of these crystals is identical with the pattern of natural material. The crystals were synthesized from aqueous alkaline sodium sulphide solutions in a graphite lined steel bomb as devised by Smith (1947) and used by Robinson (1948). Hessite was identified in the products of five bomb runs. The composition of the charges and the conditions are shown in table 1.

TABLE 1. HESSITE: BOMB CHARGES AND PRODUCTS

No.	Charges				Conditions		Products
	$Na_2S \cdot 9H_2O$	Ag	Te	H_2O	Time	Max. Temp.	
1	10 g.	0.5 g.	0.6 g.	20 cc.	3 days	400° C.	Hessite
2	40 g.	0.5 g.	0.6 g.	20 cc.	7 days	370° C.	Orthorhombic hessite
3	40 g.	0.5 g.	0.6 g.	20 cc.	7 days	360° C.	Hessite
4	50 g.	0.5 g.	0.6 g.	20 cc.	4 days	490: C.	Hessite
5	20 g.	0.75 g.	0.9 g.	10 cc.	7 days	350° C.	Isometric hessite

The bomb product identified as hessite from runs 3 and 4 was massive material with no crystal faces. These aggregates were generally fine, but some were rather large and blocky; all were covered with a yellow to purple tarnish.

The hessite present in the product of run 1 consisted of blocky, yellow or grey, metallic crystals, showing some good faces. Certain of these were long, prismatic, silvery, with prismatic striations. As good faces were not observed on these crystals, they were used only to obtain a powder diffraction pattern.

Well-formed blocky prismatic single crystals, parallel growths and aggregates, and flattened platy crystals which occurred in the product of run 2 were identified as hessite. The crystal aggregates generally displayed prismatic striations, and the crystal faces present were poorly formed but the single crystals were bounded by well defined prismatic and pyramidal forms. Most of the measured crystals of hessite came from this run.

Small cubic crystals giving the powder diffraction pattern of hessite were observed in the product of run 5. These isometric crystals are rounded with poorly defined crystal faces, and a dark purple tarnish. The principal forms are the cube, octahedron and dodecahedron.

GEOMETRICAL CRYSTALLOGRAPHY

Twenty-eight crystals of hessite from the product of run 2 were mounted for measurement on the two-circle optical goniometer. The measurements were plotted in gnomonic projection. The crystals were of two types, both orthorhombic but with different crystal forms and axial ratio. Nine crystals were distinctly type I and the measurements led to the axial ratio.

$$\text{Type I} \quad a:b:c=0.599:1:0.284$$

The observed forms together with measured and calculated ϕ and ρ angles are given in Table 2. The form (132), which is present on three crystals, shows only $(\bar{1}32)$ and $(1\bar{3}2)$ and thus suggests sphenoidal symmetry on two of these crystals, on the third all four faces were present; all other (hkl) forms show holohedral symmetry. Figure 1 is an idealized drawing of a typical crystal of type I.

Ten of the measured crystals were distinctly type II and the measurements led to the axial ratio.

$$\text{Type II} \quad a:b:c=0.776:1:0.650$$

The observed forms together with the measured and calculated ϕ and ρ angles are given in Table 3. The symmetry appears to be orthorhombic holohedral. Figure 2 is an idealized drawing of typical crystal of type II.

STRUCTURAL CRYSTALLOGRAPHY

Hessite crystals of types I and II were mounted on the Weissenberg goniometer with the prismatic axis, c, as the axis of rotation. Five crystals of type II gave rotation films with all diffractions drawn out on powder rings indicating that the prism axis is not a rational axis in the lattice or that the crystals are no longer single crystals. Sharp rotation zero and first layer Weissenberg films were obtained from a crystal of type I. Film measurements led to the following dimensions for the unit cell

$$a=16.28, \qquad b=26.68, \qquad c=7.55 \text{ A}$$[1]

with the axial ratio

$$a:b:c=0.610:1:0.283$$

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[1] Using CuKα 1.5418, mass factor 1.6602.

Table 2. Hessite Type I: Crystal Measurements

Orthorhombic, *Immm* (artificial crystals)

$a:b:c=0.599:1:0.284$; $p_0:q_0:r_0=0.474:0.284:1$

Form	Measured		Calculated		Obs.
	ϕ	ρ	ϕ	ρ	
b(010)	0°00′	90°00′	0°00′	90°00′	12
a(100)	90 00	90 00	90 00	90 00	13
m(110)	59 15	90 00	59 04½	90 00	12
n(120)	39 30	90 00	39 51	90 00	7
l(130)	28 35	90 00	29 06	90 00	7
k(150)	18 50	90 00	18 28	90 00	2
d(011)	0 00	15 45	0 00	15 51½	6
s(132)	28 35	26 00	29 06	25 59½	3
o(121)	39 50	36 00	39 51	36 30	7
r(141)	22 15	51 25	22 39	50 54½	2
(161)	18 30	61 10	15 33	60 13	1
p(211)	73 10	44 15	73 19½	44 42½	3
q(231)	48 05	51 15	48 03½	51 53	6

Table 3. Hessite Type II: Crystal Measurements

Orthorhombic (artificial crystals)

$a:b:c=0.776:1:0.650$; $p_0:q_0:r_0=0.838:0.650:1$

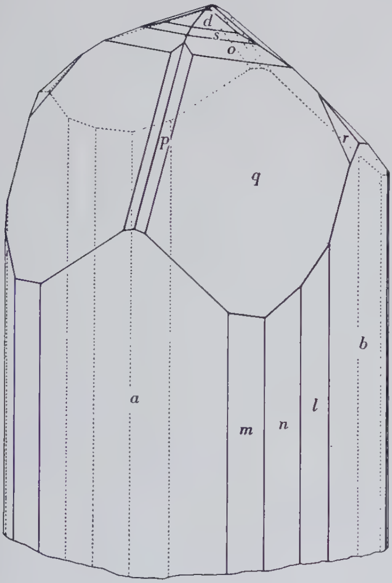
Form	Measured		Calculated		Obs.
	ϕ	ρ	ϕ	ρ	
c(001)	—	0°00′	—	0°00′	8
a(100)	90°00′	90 00	90°00′	90 00	10
l(120)	30 00	90 00	32 48	90 00	9
m(110)	52 30	90 00	52 11	90 00	4
n(320)	62 10	90 00	62 39	90 00	4
d(011)	0 00	32 50	0 00	33 01	9
p(111)	52 30	46 25	52 11	46 40	8

in good agreement with the geometrical axial ratio. The observed diffractions conform to the condition: (*hkl*) present only with $h+k+l=2n$ indicating the space group *Immm* if the crystals are holohedral. These lattice dimensions show very little similarity to those given by Tokody (1932, 1934) and Koern (1939) for hessite and artificial α-Ag_2Te

$a=6.57$ $b=6.14$ $c=6.10$ A $\beta=61°15'$ (Tokody, 1934)

$a=13.0$ $b=12.7$ $c=12.2$ A (Koern, 1939)

The *a* lengths given by Tokody and Koern are approximately $\frac{1}{4}$ and $\frac{1}{2}$, respectively, of the *b* length found for our artificial crystals of type I.



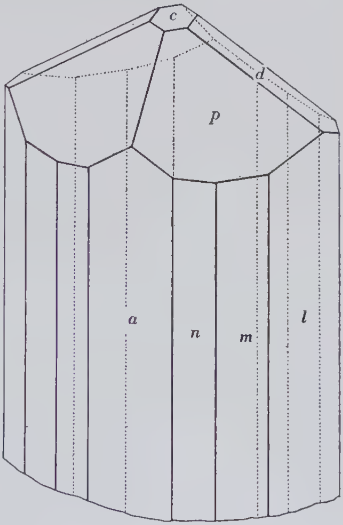


FIG. 1.—Artificial hessite crystals, type I, stable at room temperatures. Idealized crystal drawing showing the forms; *b*(010), *a*(100), *m*(110), *n*(120), *l*(130), *d*(011), *s*(132), *o*(121), *r*(141), *p*(211), *q*(231).

FIG. 2.—Artificial hessite crystal, type II, not stable at room temperatures. Idealized crystal drawing showing the forms; *c*(001), *a*(100), *l*(120), *m*(110), *n*(320), *d*(011), *p*(111).

The cubic hessite crystals from run 5 were mounted in several positions on the Weissenberg goniometer but failed to give rational rotation patterns. All three types of artificial hessite crystals give the same *x*-ray powder pattern as natural hessite. Presumably type II orthorhombic crystals and the cubic crystals represent forms stable at higher temperature which have inverted on cooling to the structure represented by type I crystals.

In Table 4 the *x*-ray powder pattern of hessite as given by Thompson

TABLE 4. HESSITE—Ag_2Te: *X*-RAY POWDER PATTERN
Orthorhombic, *Immm*; $a=16.27$, $b=26.68$, $c=7.55A$; $Z=48$

Mineral[1]			Artificial crystals			Calculated	
I	θ(Cu)	*d*(meas.)	*I*	θ(Cu)	*d*(meas.)	*hkl*	*d*(calc.)
½	6.2	7.14A	—	—	—	011	7.265A
1	9.8	4.53	—	—	—	330	4.630
						060	4.470
½	11.9	3.74	—	—	—	002	3.775
½	13.1	3.40	—	—	—	202	3.424
						071	3.402
						132	3.398
2	14.0	3.19	1	14.15	3.15	510	3.230
						271	3.139
6	14.85	3.01	5	14.95	2.99	152	3.028
						501	2.988
8	15.6	2.87	9	15.65	2.86	062	2.878
½	16.0	2.80	—	—	—	550	2.778
						402	2.767
½	16.65	2.69	½	16.75	2.67	262	2.713
						600	2.712
						422	2.709
						352	2.679
						0.10.0	2.668
						381	2.659
1	18.35	2.45	1	17.75	2.53	2.10.0	2.535
						570	2.475
						512	2.454
						123	2.445
10	19.5	2.31	10	19.65	2.29	233	2.321
						710	2.315
						660	2.315
						192	2.308
7	20.05	2.25	10	20.25	2.23	323	2.250
						730	2.249
						552	2.238
						4.10.0	2.231

Table 4 (*Continued*)

Mineral[1]			Artificial crystals			Calculated	
I	θ(Cu)	*d*(meas.)	*I*	θ(Cu)	*d*(meas.)	*hkl*	*d*(calc.)
2	20.5	2.20	8b	20.95	2.16	581	2.226
						0.12.0	2.223
						2.11.1	2.216
						602	2.202
						253	2.192
						721	2.191
						590	2.191
6	21.1	2.14	7b	21.45	2.11	392	2.142
						413	2.133
						750	2.131
						482	2.129
						671	2.121
1	22.55	2.01	1	22.45	2.02	1.11.2	2.025

Mineral[1]		Artificial crystals		Mineral		Artificial crystals	
I	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)
½	1.957A	—	—	2	1.447A	2	1.441A
½	1.926	½	1.933A	4	1.389	6	1.391
½	1.845	½	1.859	½	1.341	2b	1.342
½	1.824	—	—	2	1.305	4	1.301
1	1.771	½	1.762	1	1.278	1	1.276
½	1.734	—	—	1	1.247	2	1.248
1	1.695	½	1.678	½	1.192	½	1.192
½	1.600	—	—	—	—	½	1.000
½	1.585	½	1.578	—	—	½	0.941
½	1.544	—	—				

[1] *I* and θ from Thompson (1949).
b—broad line.

(1949) is given together with the pattern given by our artificial crystals also the indices and interplanar spacings calculated from the lattice dimensions found for hessite type I crystals. Our pattern agrees line for line with a pattern of natural hessite (Q.U. polished section TE-8, unknown locality) but the tabulated data show minor differences with Thompson's data. Some of these differences are undoubtedly due to differences in sample preparation, camera design and exposure. One or more calculated interplanar spacings agree fairly well with each measured spacing down to $d=2.0$; below this value there are many possibilities in a lattice of large unit dimensions.

X-ray powder patterns of natural hessite and artificial crystals were obtained at room temperature and at several higher temperatures in the *x*-ray powder camera designed by Buerger, Buerger & Chesley (1943) at about 125° C. the powder pattern is similar to the pattern at room temperature but with some changes in intensities. At 155° C. the pattern is much simpler in appearance and can be indexed with reference to a simple cubic lattice. The intensities measured spacings, indices and the cube edge values are given in Table 5. The indexed powder pattern leads to an average value for the cube edge.

TABLE 5. HESSITE—Ag_2Te: *X*-RAY POWDER PATTERN (at 155° C.)

Isometric P: $a=6.64$A; $Z=4$

I	θ(Cu)	d(meas.)	hkl	a
5	9.5	4.657	100	6.59
2	11.5	3.860	111	6.68
10	19.1	2.349	220	6.64
8	20.3	2.213	300, 221	6.64
3	22.5	2.009	311	6.66
3	27.5	1.665	400	6.66
2	34.8	1.347	422	6.64
			Average	6.64

$$a=6.64 \text{ A}$$

in fair agreement with the value given by Rahlfs (1936)

$$a=6.572 \text{ A}$$

COMPOSITION AND CELL CONTENT

Analyses of hessite from various localities clearly indicate a composition Ag_2Te. The measured specific gravity is 8.24 to 8.45 (Dana, 1944) 8.41 (Thompson, 1949). The unit cell volume combined with the latter value gives M = 16610. This value divided by the gram molecular weight for Ag_2Te gives $Z=48.37$. Therefore the unit cell probably contains

$$Ag_{96}Te_{48}=48[Ag_2Te]$$

This structural formula gives a calculated specific gravity of 8.35 in good agreement with the measured values. The cubic unit cell of hessite at high temperature contains 4[Ag_2Te.]

CONCLUSIONS

Orthorhombic crystals, type I, appear to be structurally identical with natural hessite. The unit cell dimensions, which differ markedly from those reported by Tokody (1932, 1934) and Koern (1939) serve to index the identical *x*-ray powder pattern obtained from the artificial crystals, artificial Ag_2Te and natural hessite. The unit cell contains 48[Ag_2Te] with calculated gravity in close agreement with measured values.

Orthorhombic crystals, type II, and cubic crystals are, at room temperature, also structurally identical with natural hessite but are clearly paramorphs of structures not stable at room temperature and pressure. Hessite, both natural and artificial, gives an isometric *x*-ray powder pattern at 155° C. which presumably corresponds to the isometric polymorph. No powder pattern was found which could be correlated with type II orthorhombic crystals.

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AN X-RAY STUDY OF THE MINERAL LIVINGSTONITE

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ABSTRACT

Livingstonite from Guerrero, Mexico is triclinic, with probable space group $P\bar{1}$. The unit cell with $a=7.65$, $b=10.82$, $c=3.99$ kX, $\alpha=99°12\frac{1}{2}'$, $\beta=102°01'$, $\gamma=73°48'$, contains $HgSb_4S_7$. Specific gravity 4.88 calculated, 5.00 measured. Cleavage (010), (100) perfect, (001) poor. The monoclinic cell of Richmond (1936) has four times the volume, with $a'=2a$, $b'=c$, $c'=2b$, $\beta=104°$.

A modern presentation of the crystallography of livingstonite—$HgSb_4S_7$—was published in the *American Mineralogist* (Richmond, 1936). Data from this paper were used in the description of livingstonite in Dana's *System of Mineralogy*, Volume 1 (1944), where it is described as monoclinic $2/m$, with unit cell dimensions a 15.14 A, b 3.98 A, c 21.60 A, β 104° and a Z factor of 4.

Some time ago Professor Peacock of the University of Toronto discovered from a study of x-ray Weissenberg photographs that livingstonite possesses triclinic symmetry. Later, S. Kaiman, one of Professor Peacock's graduate students, proved conclusively, in an unpublished work, that the mineral is triclinic. In the fall of 1949 the professor suggested that I make a formal x-ray study of this mineral, which would serve as a research problem, and fulfil partially the requirements of his x-ray crystallography course.

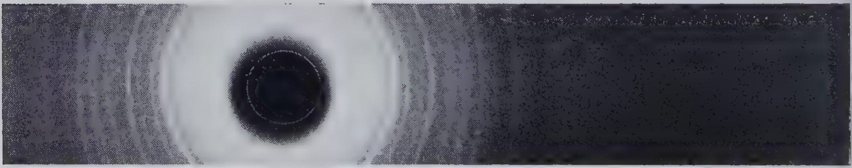

FIG. 1. X-ray powder photograph; nickel filtered copper radiation ($\lambda=1.5374$ kX) Camera radius 57.3 mm; actual size print.

A few grams of livingstonite from Guerrero, Mexico, were obtained from Mr. Frank Ebbutt, Geologist, of Toronto. No individual crystals of the material were observed, but several fine cleavage fragments were isolated. A few of these were powdered, and an x-ray powder photograph made. Figure 1 is a contact print of this photograph, taken with a Philips type camera of 57.3 mm radius, using nickel filtered copper radiation.

The spacings represented by the three most intense lines were measured, and when compared with Harcourt's values (1942), as shown in Table 1, served to identify my material with Harcourt's. The remaining spacings were measured and are recorded in Table 2.

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* Holding a National Research Council of Canada studentship.

TABLE 1. COMPARISON OF HARCOURT AND GORMAN SPACING VALUES FOR LIVINGSTONITE

Intensity	*d* Harcourt (1942)	*d* Gorman
2	3.75 kX*	3.73 kX
3	3.45	3.47
1	2.99	2.99

* Called Angstrom units in Harcourt (1942), probably kX.

TABLE 2. LIVINGSTONITE *X*-RAY POWDER DATA IN kX UNITS; Cu/Ni radiation
Triclinic; $P\bar{1}$; *a* 7.65, *b* 10.82, *c* 3.99; α 99°12′; β 102°01′; γ 73°48′

I (Cu)	*d* (meas.)	(*hkl*)	*d* (calc.)	*I* (Cu)	*d* (meas.)	(*hkl*)	*d* (calc.)	*I* (Cu)	*d* (meas.)	(*hkl*)	*d* (calc.)
4	5.14	020	5.16	½	2.49	310	2.49	3	1.875	321	1.880
1	4.86	120	4.82	3	2.42	320	2.44			$2\bar{4}0$	1.879
7	3.73	210	3.72			211	2.44			012	1.870
7	3.47	130	3.47			240	2.41	3	1.840	$\bar{4}21$	1.841
1	3.27	$0\bar{2}1$	3.28			300	2.41	5	1.729	$\bar{1}22$	1.733
10	2.99	021	2.95	6	2.27	$0\bar{4}1$	2.27	½	1.643	—	—
		$\bar{2}\bar{1}1$	3.03			330	2.27	2	1.501	—	—
		$1\bar{1}1$	3.03	2	2.03	340	2.04	1	1.423	—	—
1	2.86	$1\bar{3}0$	2.84			141	2.03	½	1.193	—	—
3	2.66	140	2.66	2	1.984	$1\bar{4}1$	1.976	½	1.097	—	—
		$2\bar{2}0$	2.66								

A cleavage fragment ½ mm in length was isolated, and mounted on an *x*-ray goniometer so that the cleavage edge between two good cleavage faces was parallel to the axis of rotation of the goniometer head. Rotation, zero layer and first layer Weissenberg photographs were then taken.

The cleavage edge was arbitrarily chosen as the direction of the *c* axis, hence the rotation photograph gave *c*. The zero layer Weissenberg gave *d*(100) and *d*(010), and γ^*. The first layer Weissenberg gave the horizontal offset of the reciprocal lattice, measured by the rectangular co-ordinates *x* and *y*, which are functions of α^* and β^*. The lengths *x* and *y* were measured in millimeters. From these six measurements the reciprocal lattice elements were calculated. Standard formulae were then used to convert these reciprocal lattice values to those of the direct lattice.

Thus from one setting of the cleavage fragment, the direct lattice elements were obtained. The elements *a*, *b*, α and β are dependent on the measurement of *x* and *y* and are less accurate than *c* and γ which are independent of graphical measurement. In Table 3 the lattice constants used in the calculations are given. Table 2 shows the lattice constants modified to conform with the accuracy of measurements.

TABLE 3. LIVINGSTONE LATTICE CONSTANTS USED IN CALCULATION

Measured		Reciprocal Lattice		Direct Lattice	
c	3.990 kX	a*	0.13819	a	7.650 kX
d(100)	7.236 kX	b*	0.09685	b	10.817 kX
d(010)	10.326 kX	c*	0.25776	c	3.990 kX
γ^*	104°44½′	α^*	83°46′	α	99°12½′
x	8.2 mm.	β^*	80°04′	β	102°01′
y	4.3 mm.	γ^*	104°44½′	γ	73°48′

The unit cell was chosen with a less than b and α and β both obtuse. This is the modern setting for triclinic crystals, which restricts the slope of the base (001) to the front and the right of the front pinacoid. Fortuitously c is less than a or b.

The cell constants having been found, the next task included calculating the spacings of the planes whose spacings had been obtained from the powder photograph. This involved the use of the "Bravais Sphere," and a graphical method of determining the spacings of lattice planes as proposed by Peacock (1938). The measured spacings were used to find, on a gnomonic projection, planes with indices that might possibly be responsible for such spacings. Then these indices were used in a formula involving the lattice constants to calculate spacings. When the calculated spacings agreed with the observed spacings within 2/10 of θ angle, then the plane used in the calculation was recorded as a plane responsible for a diffraction line on the powder photograph. Table 2 gives the complete x-ray data for livingstonite, with observed intensities for copper radiation based on a 10-1 scale, and measured and calculated planar spacings with their corresponding indices.

A comparison of Richmond's cell to the present author's is given in Table 4.

TABLE 4. COMPARISON OF RICHMOND AND GORMAN CELLS FOR LIVINGSTONITE

Richmond		Gorman			
a	15.14 kX*	a	7.65 kX	α	99°12′
b	3.98	b	10.82	β	102°01′
c	21.60	c	3.99	γ	73°48′
β	104°				

* Given by Richmond as A; probably kX.

From Table 4 it may be seen that two of the linear elements of Richmond are halved by the present author, and that *b* and *c* are interchanged. Otherwise, the similarity of the cells is apparent. From his unit cell dimensions, Richmond has calculated that the *Z* factor must be 4, which means that the cell contents are 4 [$HgSb_4S_7$]. Since the new cell volume is approximately $\frac{1}{4}$ that of Richmond's, one would expect the structural formula to be 1 [$HgSb_4S_7$].

The new elements give a cell volume of 308.27kX^3. The calculated density using this value and the structural formula 1 [$HgSb_4S_7$] is 4.88 grams per cubic centimeter, which is in fair agreement with 5.00 as measured on the Berman balance. Richmond's calculated value was 4.79 as against 5.00 measured by Frondel (Dana, 1944). The agreement is sufficiently close to warrant the acceptance of the structural formula 1 [$HgSb_4S_7$].

The change in the orientation of the cell, apart from the modification of monoclinic to triclinic, necessitates a renaming of the cleavage directions. Nevertheless, there is little change in the calculated angle between the two best cleavages, recorded by Richmond as 104° and here as 104°21′. Table 5 shows the comparison between cleavages in the old and new setting.

TABLE 5. CLEAVAGES OF LIVINGSTONITE

Richmond		Gorman	
(001)	perfect	(010)	perfect
(100)	perfect	(100)	perfect
(010)	poor	(001)	poor

The space group $P\bar{1}$ was chosen in preference to $P1$, merely because the former is common, the latter rare.

I would like to express my appreciation to the National Research Council of Canada for their financial aid, and for their permission to publish this paper. I would like also to record my thanks to Dr. E. W. Nuffield of the University of Toronto for his critical reading of the manuscript.

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OBSERVATIONS ON CONICHALCITE, CORNWALLITE, EUCHROITE LIROCONITE AND OLIVENITE

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Abstract

Conichalcite, is orthorhombic, *Pnam*; the unit cell with $a=7.40$, $b=9.21$, $c=5.84$A contains $4[CuCaAsO_4(OH)]$, calculated specific gravity, 4.33. Higginsite and some specimens of erinite give the same *x*-ray powder pattern as conichalcite.

Cornwallite is monoclinic, $P2_1/a$; the unit cell with $a=17.61$, $b=5.81$, $c=4.60$A, $\beta=92°15'$ contains $2[Cu_5(AsO_4)_2\cdot(OH)_4]$. Specific gravity 4.68 (calc.) 4.17 (Church, 1868). Several specimens of erinite give the same *x*-ray powder pattern. The powder patterns indicate the existence of an isostructural series with pseudomalachite.

Euchroite is orthorhombic, $P2_12_12_1$. The unit cell with $a=10.07$, $b=10.52$, $c=6.12$A contains $4[Cu_2AsO_4(OH)\cdot 3H_2O]$, calculated specific gravity 3.45, measured 3.41. Optical orientation $Z=b$, optic plane (001).

Liroconite is monoclinic, $I2/a$. The unit cell with $a=12.70$, $b=7.57$, $c=9.88$A, $\beta=91°23'$ contains $4[Cu_2Al(As,P)O_4(OH)_4\cdot 4H_2O]$ calculated specific gravity 2.95, measured 3.01.

Olivenite is orthorhombic, *Pmnm*. The unit cell with $a=8.22$, $b=8.64$, $c=5.95$A, contains $4[Cu_2AsO_4(OH)]$ with calculated specific gravity 4.45, measured 4.378. Leucochalcite from Wilhelmine mine Spessart Germany (type locality) and from Majuba Hill, Nevada give the same *x*-ray powder pattern.

In the course of a study of natural hydrous and basic copper phosphates and arsenates a large number of museum specimens of these interesting minerals have been examined. *X*-ray powder diffraction patterns of all specimens have revealed numerous misidentifications and has indicated that a number of the older mineral names apply to the same mineral. This was particularly true of the green copper minerals occurring in finely crystalline and botryoidal crusts. The results of this study of pseudomalachite, cornetite and clinoclasite have already appeared (Berry, 1950; Palache & Berry 1946). In the present paper the results are given for several of the arsenates.

The writer wishes to acknowledge the courtesy and cooperation of the following men and institutions in providing the materials used for study; Dr. F. A. Bannister, British Museum of Natural History (BM); Dr. W. F. Foshag and Dr. G. Switzer, United States National Museum (USNM); Dr. C. Frondel, Harvard University Mineralogical Museum (HM); Dr. F. Pough, American Museum of Natural History (AMNH); Dr. V. B. Meen, Royal Ontario Museum of Mineralogy (ROM); Dr. H. Winchell, Yale University Museum (YM); Hatfield Goudey, Yerrington, Nevada; and Dr. C. Milton, United States Geological Survey for loaning films of numerous specimens for comparison. The writer also acknowledges the assistance of several former students in much of the formal work. A. R. Graham assisted with conichalcite, H. R. Steacy with euchroite (Berry & Steacy, 1947), and T. Davies with liroconite (Berry

& Davies, 1947). The data on conichalcite have also appeared in abstract (Berry 1948).

CONICHALCITE AND "HIGGINSITE"

Conichalcite was first described by Breithaupt (1849) with analyses by Fritsche on reniform and massive material from Spain. The mineral was later recognized and analysed by Hillebrand (1884) from Tintic, Utah. Higginsite was described by Palache & Shannon (1920) as well crystallized material from the Higgins mine, Bisbee, Arizona. Strunz (1939) gives unit cell dimensions for higginsite and finds that conichalcite and higginsite give almost identical x-ray powder patterns. Independently, Richmond (1940) determined the unit cell dimensions and space group for higginsite.

In the present study numerous museum specimens labelled conichalcite and erinite were examined. All the conichalcite specimens and some of the erinite specimens gave an *x*-ray powder pattern identical with that from a crystal of higginsite, kindly provided from the type material by Professor C. Frondel of Harvard University. This pattern was obtained from the following specimens:

Conichalcite: Simon Mine, Mineral Co., Nevada (HM, 86387); Tintic, Utah (QM, M8282, A561-2); Copperopolis, Tintic (ROM, M20135); Tintic (ROM, M20128); Calavada mine, Yerrington, Nevada (HM, Goudey); Napa Valley, California (USNM, 13584).

"Higginsite": Higgins mine, Bisbee, Arizona (HM, 92923, type material).

"Erinite": Tintic, Utah (ROM, E3001), (ROM, M6761); Redruth Cornwall (USNM, R5377); Utah (AMNH, 15311)

"Mixite"; Utah (ROM, M18910)

One of these specimens labelled "erinite" (ROM, E3001) yielded a small crystal from a radiating group suitable for study on the Weissenberg goniometer. The films yielded the following lattice dimensions:

$a=7.40$ $b=9.26$ $c=5.87$ A (A. R. Graham)

$a=7.42$ $b=9.21$ $c=5.84$ A (Strunz, 1939)[1]

$a=7.43$ $b=9.22$ $c=5.86$ A (Richmond, 1940)[1]

in close agreement with the values given by Strunz and Richmond. In order to obtain satisfactory agreement between measured and calculated spacings in the *x*-ray powder pattern (Table 1) the following lattice dimensions were chosen:

$$a=7.40, \quad b=9.21, \quad c=5.84 \text{ A}$$

Three of the specimens examined here (ROM, E3001, M20135; HM, 86387), including that used for single crystal study, gave an *x*-ray powder pattern slightly contracted compared to the others, indicating a slightly larger unit cell. The pattern given in table 1 represents that given by "higginsite" and most of the conichalcite specimens. The variation may be due to substitution of phosphorus for arsenic but a further x-ray study of carefully analysed materials would be necessary to establish this.

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[1] Converted to new Angstrom wave lengths.

Composition and cell content. Strunz (1939) and Richmond (1940) show clearly that the unit cell content of higginsite is 4[$CuCaAsO_4(OH)$] with calculated specific gravity, 4.33, in good agreement with the measured value 4.33. The available analyses of conichalcite by Fritsche (Breithaupt, 1849, Cordoba, Spain), Hillebrand (1884, Tintic, Utah) and Michel (1909, Maya-Tass, Akmolinsk, Siberia) differ notably in the water content of 5.61, 5.52, and 5.15 per cent respectively compared with 3.41 per cent given by Shannon (Palache & Shannon, 1920) for higginsite. Very finely crystalline materials of this type do show a higher water content than well formed crystals, this has been already noted in a survey of pseudomalachite analyses (Berry, 1950).

TABLE 1. CONICHALCITE—$CuCaAsO_4(OH)$: X-RAY POWDER PATTERN
Orthorhombic, *Pnam*, $a=7.40$, $b=9.21$, $c=5.84$A; $Z=4$

I	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)
3	5.73A	110	5.769A	3	2.36A	310	2.383A	5	1.822A	331	1.826A
2	4.91	011	4.932			230	2.363	6	1.720	203	1.723
5	4.10	111	4.104			122	2.340			420	1.716
1	3.93	120	3.910	2	2.29	202	2.292	7	1.609	223	1.614
2	3.70	200	3.700	1	2.21	212	2.224			332	1.606
2	3.42	210	3.433			231	2.190			133	1.605
1	3.25	121	3.249	4	2.05	222	2.052	2	1.560	402	1.562
9	3.14	201	3.125			132	2.034	3	1.527	060	1.535
10	2.84	220	2.884	½	1.963	240	1.955			152	1.524
		130	2.836	4	1.843	241	1.854				
10	2.59	112	2.605			400	1.850				
		221	2.586			312	1.846				
5	2.56	131	2.551			113	1.845				
1	2.46	022	2.466			232	1.837				

I	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)
½	1.479A	4	1.301A	1	1.115A	½	1.002A	½	0.814
1	1.462	1	1.274	3	1.099	½	0.989	½	0.803
½	1.442	½	1.246	½	1.079	½	0.926	1	0.794
½	1.415	3	1.211	½	1.064	½b	0.906		
2	1.381	½	1.189	½	1.039	1	0.836		
½	1.371	½	1.145	½	1.018	1	0.826		

b—broad line.

The name conichalcite is preferred to higginsite for this mineral since it has priority and the analyses lead to a cell content which is essentially the same as that found for higginsite, the well crystallized material.

CORNWALLITE AND "ERINITE"

Cornwallite, erinite and conichalcite are names which have been associated with green botryoidal crusts which are generally hydrous copper arsenates in composition. Conichalcite, discussed briefly in the first section of this paper, contains calcium in addition to copper. Eleven specimens labelled erinite were available for study; four gave the *x*-ray powder pattern of conichalcite and seven gave an *x*-ray powder pattern similar to the pattern given by most of the cornwallite specimens. The following specimens are referred to in this section:

1. "Erinite" (YM, Brush Coll., 3064) Mammoth mine, Tintic District, Utah. Dark green, fine crystalline crust on olivenite crystals and massive enargite.
2. "Erinite" (YM, 7310) Mammoth mine, Tintic District, Utah. Dark green, fine crystalline crust coating and replacing azurite crystals.
3. Cornwallite (BM, AG, 2) Cornwall. Green, thin botryoidal coating.
4. "Erinite" (BM, 39508) Ireland, "near type" material given to the British Museum by Haidinger in 1858. Light green, fine crystalline radiating crust.
5. Cornwallite (USNM, 105188) Tintic, Utah. *X*-ray powder pattern by United States Geological Survey.[1]
6. Cornwallite (Hatfield Goudey) Majuba Hill, Nevada. Light green crust.
7. "Erinite" (USNM, C4200) Eureka, Nevada. *X*-ray powder pattern by United States Geological Survey.[1]
8. "Erinite" (USNM, 48112) Tintic, Utah, Green crust with clinoclasite.
9. "Erinite" (HM, Holden Collection) Centennial Eureka mine, Tintic, Utah. Thin dark green fine crystalline coating over enargite in vugs, some later nodules of malachite.
10. Cornwallite (BM, 35618) St. Day United mines, Gwennap, Cornwall, Green cryptocrystalline material.
11. Cornwallite (BM, 55559) St. Day United Mines, Gwennap, Cornwall. Fine crystalline green material.
12. Cornwallite (USNM, R5568) Cornwall, England. Green cryptocrystalline nodules with olivenite.
13. "Erinite" (BM, 61333) Gunnislake mine, Calstock, Cornwall, Light green globular aggregate with limonite.
14. Cornwallite (BM, 36469) Wheal Unity, Gwennap, Cornwall. Green globular coating.

The *x*-ray powder patterns given by these materials will be discussed later.

Crystallography. The specimens all showed radiating botryoidal crusts up to 1 mm. thick. On material 1 the crust was distinctly crystalline under the binocular microscope (20×), after several trials a fragment was detached from the surface which showed two poor reflections on the optical goniometer. The crystal fragment was adjusted about the edge between these two faces. Rotation, zero and first layer Weissenberg films showed the crystal to be monoclinic with *b* as the rotation axis. Precession films about an axis parallel to each face and perpendicular to the b axis showed the two faces to be $c(001)$ and $a(100)$. The films yielded the following measured lattice spacings

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[1] Compared through courtesy of Dr. Charles Milton, personal communication February 11, 1948.

$$d(100)=17.6 \quad d(010)=5.81, \quad d(001)=4.60 \text{ A}, \quad \beta^*=87°45'$$

giving monoclinic lattice dimensions

$$a=17.61, \quad b=5.81, \quad c=4.60 \text{ A}, \quad \beta=92°15'$$

These dimensions are similar, but all slightly larger, than those found for pseudomalachite:

$$a=17.06, \quad b=5.76, \quad c=4.49 \text{ A}, \quad \beta=91°02' \text{ (Berry, 1950)}$$

The observed diffractions conform to the conditions: (hkl) present in all orders, $(h0l)$ present only with $h=2n$, $(0k0)$ present with $k=2n$; these criteria are characteristic of the space group $C_{2h}^5-P2_1/a$. This same space group was derived for pseudomalachite.

Composition and cell content. The unit cell dimensions for cornwallite combined with the measured specific gravity 4.17 (Church, 1868) give the molecular weight of the unit cell M = 1182.4. Table 2 shows the analysis of cornwallite by Church (1868) and the atomic contents of the unit cell using the above value for M.

TABLE 2. CORNWALLITE: ANALYSIS AND CELL CONTENT

	1	2		3	4	5
CuO	59.95	8.80	Cu	8.80	10	59.94
As_2O_5	30.47	1.55	As	3.10	4	34.63
P_2O_5	2.71	0.23	P	0.46		—
H_2O	8.23	5.40	H	10.80	8	5.43
	101.36		O	23.10	24	100.00

1. Cornwall; anal. Church (1868). 2. Unit Cell content. 3. Number of atoms in the unit cell. 4. Ideal cell content $2[Cu_5(AsO_4)_2(OH)_4]$. 5. Composition for ideal cell content.

The unit cell content agrees rather poorly with the ideal cell content suggested by the similarity of this mineral with pseudomalachite, $2[Cu_5(PO_4)_2(OH)_4]$. The number for H is high and the numbers of atoms of Cu and As are low. The divergence from the ideal is similar to that found for many analyses of pseudomalachite. It was pointed out in the discussion of the analyses of pseudomalachite (Berry, 1950) that good agreement with the ideal cell content was found for analyses of coarsely crystalline materials; while colloform or botryoidal finely crystalline materials possess a higher water content. Cornwallite or erinite has never been reported as occurring in coarsely crystalline form.

The ideal cell content $2[Cu_5(AsO_4)_2(OH)_4]$ gives a calculated specific gravity 4.68 which is considerably higher than the measured values 4.17 (cornwallite, Church, 1868) 4.043 (erinite, Turner, 1828). The analysis shows substitution of phosphorus for arsenic to the extent of one-half atom which would account for part of the discrepancy. The measured value is probably low since the material is a botryoidal crust on olivenite. In the study of pseudomalachite colloform and cryptocrystalline materials were found to give distinctly lower values for the specific gravity. Our specimens all consisted of colloform crusts over olivenite, enargite or altered rock, consequently visibly pure fragments large enough for a Berman balance determination were not obtainable.

The calculated specific gravity, 4.68, for $2[Cu\ (AsO_4)_2(OH)_4]$, is not inconsistent when compared with the calculated values, 4.34 for pseudomalachite $2[Cu_5(PO_4)_2(OH)_4]$; 4.45 for olivenite $4[Cu_2AsO_4(OH)]$; and 3.94 for libethenite $4[Cu_2PO_4(OH)]$.

In table 3 the available analyses of cornwallite are reduced to atomic proportions on the basis of 24 atoms of oxygen. The numbers of atoms show a similar variation to that shown by pseudomalachite analyses and the average numbers of atoms show reasonable agreement with the ideal numbers for $2[Cu_5(AsO_4)_2(OH)_4]$ except in the number for hydrogen. Analysis 1 by Turner (1828) is the only one which shows good agreement with the ideal composition.

TABLE 3. CORNWALLITE AND "ERINITE"; ANALYSES REDUCED TO 24 ATOMS OF OXYGEN

	1	2	3	4	5	6	A	B
Cu	10.20	9.53	9.36	9.37	9.15	9.04	9.44	10
As	4.00	3.75	3.76	3.66	3.22	3.46	3.73	4
P	—	—	.02	—	.48	—		
H	7.60	10.20	10.36	10.96	11.20	12.60	10.49	8
O	24.00	24.00	24.00	24.00	24.00	24.00	24.00	24

1. "Erinite," Cornwall, anal. Turner (1828) "approximate analysis" includes Al_2O_3—1.77. 2 and 4. "Erinite" Mammoth mine, Tintic, Utah. anal. Pearce (1886) analysis 2 includes Fe_2O_3—0.85, CaO—0.43, SO_3 trace. 3 and 6. "Erinite," American Eagle mine, Tintic, Utah, anal. Hillebrand (1888) analysis 3 includes ZnO—1.06, CaO—0.32, Fe_2O_3—0.14 and anslysis 6 includes ZnO—0.59, CaO—0.51, Fe_2O_3—0.20. 5. Cornwallite Cornwall, anal. Church (1868).

Hillebrand (1888) states, with reference to analyses 3 and 6 table 3, that if the material is dried over H_2SO_4 instead of in air the water content would be 6.08 per cent and 5.93 per cent instead of 7.22 and 9.15 respectively and if the lower water content is used the ratio $CuO:As_2O_5:H_2O$ is much closer to that indicated by Turner's analysis (5:1:2 as also required by the structural formula proposed here). These percentages of water are not far from the value 5.43 required by the structural formula. These observations by Hillebrand lend support to the conclusion, indicated by the present studies on cornwallite and pseudomalachite, that the colloform and botryoidal materials contain non-crystalline inclusions which result in higher water content, lower arsenic or phosphorus and copper content and lower specific gravity.

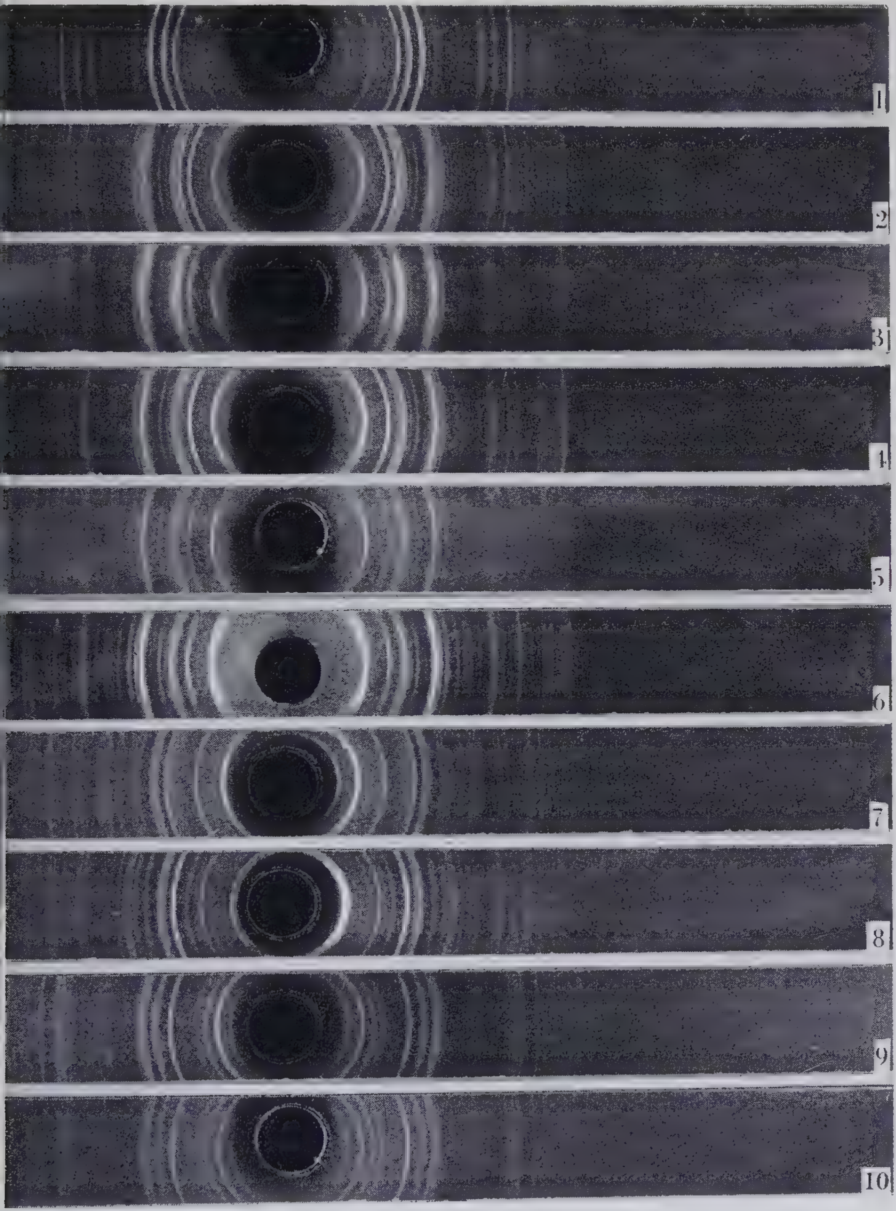
X-ray powder pattern. Materials 1–9 give exactly identical *x*-ray powder diffraction patterns (Figure 2). The pattern for material 10 (Figure 3) is diffuse but otherwise identical with material 1. The pattern for material 1 (used for single crystal study) is reproduced in Figure 2. The estimated intensities and measured spacings from this pattern are listed in table 4 together with the indices and calculated spacings as far as $d=2.22$ A. Materials 11 (Figure 4) and 12 give patterns which differ slightly in the relative intensities and spacings of the lines from the pattern of material 1. Materials 13 (Figure 5) and 14 give patterns closely similar to the pattern of pseudomalachite (Figure 6). The pattern for material 14 is very diffuse. The observed intensities and measured spacings for materials 1, 11, 13, 14 and pseudomalachite (Berry, 1950)

TABLE 4. CORNWALLITE—$Cu_5(AsO_4)_2OH_4$: *X*-RAY POWDER PATTERN*
Monoclinic, $P2_1/a$; $a=17.61$, $b=5.81$, $c=4.60$A, $\beta=92°15'$; $Z=2$

I	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)
2	5.40A	110	5.518A	2	2.88A	020	2.904A	9	2.40A	121	2.426A
5	4.82	210	4.849			120	2.866			420	2.424
6	4.60	001	4.600	4	2.73	220	2.759			$\bar{2}21$	2.379
9	3.52	$\bar{1}11$	3.555			411	2.748	4	2.34	221	2.353
		111	3.512	½	2.59	610	2.619	6	2.29	$\bar{6}11$	2.311
		410	3.508			320	2.603			710	2.307
½	3.34	$\bar{2}11$	3.374	1	2.54	$\bar{5}11$	2.559			002	2.300
		211	3.302			$\bar{6}01$	2.518			$\bar{3}21$	2.283
10	3.20	$\bar{4}01$	3.244	8	2.47	511	2.481	1	2.22	321	2.249
8	3.10	401	3.119			021	2.456			611	2.242
		$\bar{3}11$	3.115			$\bar{1}21$	2.440			520	2.241
6	3.00	311	3.031			601	2.430			800	2.200
		510	3.011								

I	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)
2	2.17A	2	1.608A	5	1.341A	½	1.089A	½	0.884A
1	2.11	2	1.580	½	1.306	½	1.065	½	0.850
½	2.06	1	1.547	½	1.279	½	1.038	½	0.835
3	1.873	3	1.521	1	1.247	1	1.022	½	0.820
2	1.828	½	1.501	½	1.234	½	0.999	½	0.815
1	1.791	½	1.486	1	1.218	½	0.991	½	0.805
4	1.765	3	1.457	1	1.208	2	0.938	½	0.803
3	1.731	3	1.425	1	1.151	½	0.929	½	0.791
1	1.678	3	1.399	½	1.137	½	0.910		
6	1.645	3	1.368	½	1.114	½	0.895		

* Material 1.



FIGS. 1–10.—*X*-ray powder photographs with Cu/Ni radiation; Camera radius $90/\pi$ mm. ($1°\theta = 1$ mm. on film); full size reproduction of contact prints. Fig. 1. Conichalcite. Fig. 2. "Erinite" = cornwallite, Mammoth mine, Tintic District, Utah (material 1). Fig. 3. Cornwallite, St. Day United mines, Gwennap, Cornwall (material 10). Fig. 4. Cornwallite, St. Day United mines, Gwennap, Cornwall (material 11). Fig. 5. "Erinite," Gunnislake mine, Calstock, Cornwall (material 13). Fig. 6. Pseudomalachite, Nassau, Germany (material 9 in Berry, 1950). Fig. 7. Euchroite, Libethen, Hungary. Fig. 8. Liroconite, Cornwall. Fig. 9. Olivenite. Fig. 10. "Leucochalcite" = olivenite, Majuba Hill, Nevada.

Table 5. Cornwallite—Pseudomalachite: *X*-Ray Powder Data

Material 1		Material 11		Material 13		Material 14		Pseudomalachite	
I	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)
2	5.40A	1	5.47A		—				—
5	4.82	2	4.77		—			½	4.75A
6	4.60	8	4.53	10	4.52A	10	4.53A	10	4.48A
9	3.52	7	3.51	6	3.50	3	3.46	5	3.46
½	3.34	½	3.31					½	3.27
10	3.20	5	3.18	7	3.14			2	3.12
8	3.10	6	3.08	5	3.03	7b	3.06	{4 2	3.09 3.04
6	3.00	6	3.00	5	2.99			4	2.97
	—		—		—			3	2.93
2	2.88	2	2.88	1	2.86			1	2.85
4	2.73	3	2.72	3	2.74			3	2.72
½	2.59	1	2.57	1	2.56			1	2.56
1	2.54	½	2.51		—				—
8	2.47	4	2.45	9	2.44			6	2.42
9	2.40	10	2.40	8	2.39	10b	2.40	8	2.39
4	2.34	4	2.33	4	2.33			5	2.32
6	2.29	5	2.28	5	2.25	2	2.24	5	2.23
1	2.22	½	2.22	½	2.21			1	2.19
2	2.17	½	2.17	½	2.15			1	2.12
1	2.11	3	2.10	3	2.10	1	2.10	3	2.09
½	2.06	½	2.04	2	2.01	2	2.01	1	2.01
	—	½	1.993	½	1.955			2	1.963
	—		—		—			1	1.939
3	1.873	3	1.870	1	1.864			2	1.854
2	1.828	½	1.817	½	1.815				—
1	1.791	1	1.781	1	1.770	3	1.752	4	1.763
4	1.765	5	1.755	4	1.738			5	1.728
3	1.731	1	1.722		—				—
1	1.678	2	1.678	1	1.674			2	1.670
6	1.645	4	1.629	2	1.635			1	1.624
2	1.608	1	1.598		—	2	1.580	2	1.597
2	1.580	3	1.570	3	1.569			4	1.559
1	1.547	1	1.542	1	1.536			2	1.527
3	1.521	2	1.517	3	1.504			2	1.498
½	1.501		—		—	½	1.492	2	1.491
½	1.486		—	½	1.470			1	1.462
3	1.457	3	1.449	3	1.438	½	1.431	3	1.431
3	1.425	{2 3	1.435} 1.414}		—			3	1.419
3	1.399	½	1.399	2	1.399	1	1.404	4	1.392
	—	1	1.372	1	1.370			1	1.365
3	1.368	½	1.358	2	1.353			2	1.350
5	1.341	6	1.337	3	1.333	3b	1.337	3	1.335
	—		—		—			2	1.319
½	1.306		—		—			3	1.310

b—broad line.

are given in table 5. The spacings and intensities show a gradual change from material 1, cornwallite, to pseudomalachite; the spacings decrease, some intensities increase and some decrease. The pattern given in the ASTM card files for material from Gunnislake, Cornwall appears to be very close to the pattern of material 14 and pseudomalachite.

Optical data. New determinations of optical data have not been made largely because the indices lie above the range of oils readily available. Data on material 8 (USNM 48112) are given by Larsen (1921) who also gives data for "cornwallite (USNM, Roebling Coll.) Cornwall, England" which probably applies to material 12.

	*n*X	*n*Y	*n*Z
"erinite" (material 8)	1.820	1.86	1.88
cornwallite (material 12?)	1.81	1.815	1.85
pseudomalachite (Barth & Berman, 1930)	1.785	1.850	1.862

The available optical data show roughly a gradation from cornwallite to pseudomalachite. The indices for cornwallite are probably inaccurate since the mineral decomposes in oils containing As_2S_3 and S, with evolution of gas and development of crystals (Larsen, 1921).

The *x*-ray data clearly indicate that cornwallite and erinite are the same mineral but show a gradation to pseudomalachite, due probably to phosphorus substituting for arsenic. This is supported by the meagre optical data. The name erinite has priority and the composition ascribed to erinite from Turner's analysis is the same as the structural formula deduced here. Material labelled erinite (material 4) by Haidinger is identical with cornwallite. However the name erinite has been misused for chalcophyllite by some early authors and the mineral has been shown to be a Cornish mineral rather than from Ireland as stated by Haidinger, (Dana, 1892). It is proposed here that the name cornwallite be retained for this mineral.

EUCHROITE

Euchroite was first named by Breithaupt (1823) and described by Haidinger (1825) from Libethen, Hungary. The mineral has not been reported from any other locality. Two specimens, ROM M15178 and USNM 47056 from Libethen, Hungary were used in this study. Well formed green crystals, one to five millimeters in length are implanted in rusty sericite schist. Three specific gravity determinations were made on a clean crystal weighing 28 mg., using the Berman balance. The average compares closely with other determinations.

3.41 (Steacy, 1946)
3.389 (Haidinger, 1825)
3.42 (Church, 1895)
3.44 (Frondel, priv. comm.)

Structural crystallography. A crystal about one millimeter in length, was easily detached from the first specimen referred to above. Using unfiltered copper radiation, rotation and Weissenberg films were made about the c (axis of elongation) and b axes, yielding the following orthorhombic lattice dimensions:

$$a=10.07, \qquad b=10.52, \qquad c=6.12 \text{ A}$$

The observed diffractions conform to the conditions (hkl), $(0kl)$, $(h0l)$ and $(hk0)$ all present, $(h00)$ present with $h=2n$, $(0k0)$ present with $k=2n$, $(00l)$ present with $l=2n$. These criteria are characteristic of the space group.

$$D_2^4=P2_12_12_1$$

Geometrical crystallography. On crystals of euchroite from Libethen, Haidinger (1825) obtained orthorhombic elements:

$$a:b:c=0.6088:1:1.0379$$

Haidinger's elements agree closely with the structural elements in a different setting

$b:c:a=0.9635:1:0.5866$ (Haidinger, 1825)

$a:b:c=0.057\ :1:0.582$ (structural elements)

TABLE 6. EUCHROITE: OBSERVED FORMS

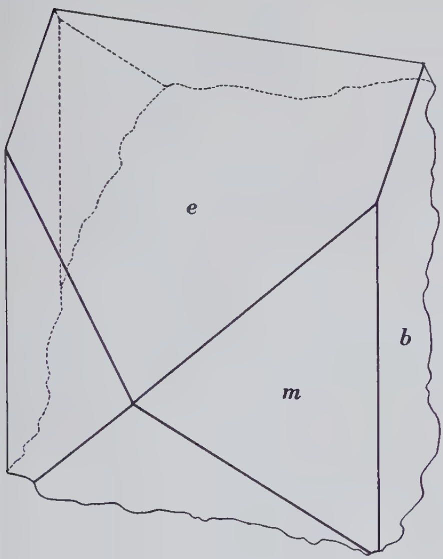
Haidinger (1825)	Steacy (1946)	Structure
$c(001)$	$b(010)$	$b(010)$
$b(010)$		$a(100)$
$n(011)$	$m(110)$	$m(110)$
$e(101)$		$n(011)$
$d(102)$		$d(021)$
$m(110)$	$e(101)$	$e(101)$
$s(230)$		$s(302)$
$l(120)$		$l(201)$

Measurement of three crystals (Figure 11) from our specimens revealed only the forms $b(010)$, $m(110)$ and $e(101)$ with measured angles in fair agreement with angles calculated from the structural elements (Table 7). The faces of $e(101)$ on our crystals gave poor reflections therefore the observed angles $(101)\wedge(\bar{1}01)$ are rough measurements.

Composition and cell content. The volume of the unit cell of euchroite together with the measured specific gravity 3.41 gives the molecular weight of the unit cell M=1331.7. Using this value for M, the five available analyses of euchroite give the unit cell contents shown in Table 8.

Table 7. Euchroite: Observed and Calculated Angles

Angle	Observed		Calculated (Structure)	Calculated (Haidinger)
	Range	Average		
(101)∧(1̄01)	63°26′–64°26′ (4)	63°56′	62°36′	62°40′
(010)∧(110)	46 08 –46 35′ (6)	46 19	46 15	46 04



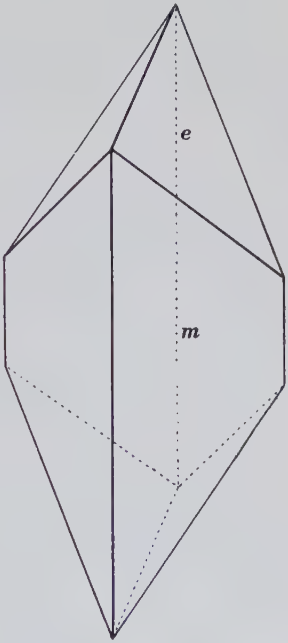


Fig. 11.—Euchroite, Libethen, Hungary. Crystal drawing showing the forms *b*(010), *m*(110), *e*(101).

Fig. 12. Liroconite, Cornwall. Crystal drawing showing the forms *m*(110), *e*(011).

The numbers of atoms agree closely with the average numbers (A) and the ideal number (B) for the cell content represented by the formula.

$$8CuO \cdot 2As_2O_5 \cdot 14H_2O = 4[Cu_2AsO_4(OH) \cdot 3H_2O]$$

This formula yields the calculated specific gravity 3.45, in close agreement with the measured values.

X-ray powder pattern. One of the crystals of euchroite from the same specimen as the crystal used for Weissenberg study was crushed, and the *x*-ray film obtained from this powder with filtered CuK radiation is re-

Table 8. Euchroite: Analysis and Cell Content

	1	2	3	4	5	A	B
CuO	47.85	46.97	46.99	48.09	47.26	47.45	47.10
As_2O_5	33.02	34.42	32.42	33.22	30.90	33.09	34.20
P_2O_5	—	—	—	—	1.48		
H_2O	18.80	19.31	19.31	18.39	19.28	19.02	18.70
	99.67	100.70	98.72	99.70	98.92	100.56	100.00
Cu	8.02	7.88	7.89	8.07	7.93	7.96	8
As	3.82	4.00	3.76	3.86	3.90*	3.87	4
H	27.86	28.62	28.62	27.26	28.28	28.19	28
O	31.50	32.19	31.60	31.35	31.57	31.64	32

1. Libethen, Turner, (1825). 2–3 Libethen, Kühn, (1844) (3 includes CaO 1.12). 4. Libethen, Wöhler, (1844). 5. Libethen Church, (1895). A. Average composition and cell content. B. Ideal composition and cell content for $4[Cu_2AsO_4(OH) \cdot 3H_2O]$.

* Includes P=0.28.

produced in figure 7. The estimated intensities and interplanar spacings obtained from this film are given in Table 9. The measured spacings each agree well with one or more spacings calculated from the structural lattice dimensions. The pattern agrees closely with that recorded in the A.S.T.M. card file.

Optical properties. A typical crystal of euchroite was mounted on the universal stage for redetermination of the optical orientation. 2V was also measured. The mineral is biaxial, positive with $Z=b$ the optic plane parallel to (001) and $2V=31°$.

Liroconite

Liroconite is a hydrous arsenate of copper and aluminum which occurs with other arsenates in vugs and cavities in the rusty wall rock of copper deposits in Cornwall, England. On the four specimens available for this study (UT A/2138, USNM, 103848, HM 96238, AMNH*) the crystals occur singly and as sub-parallel groups, also with a granular texture. Individual crystals range from 0.3 to 7 mm. in length, they are sky blue to verdigris-green in colour. The fourth specimen, from Wheal Unity, Gwennap, Cornwall shows both a sky blue crystalline aggregate of liroconite and well developed green crystals with azurite, malachite and clinoclasite on cuprite. These specimens including material of both

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* Obtained in England in 1948 from George Croker Fox collection and kindly loaned for study by Dr. Fred Pough, American Museum of Natural History.

TABLE 9. EUCHROITE: $Cu_2AsO_4(OH)\cdot 3H_2O$: X-RAY POWDER PATTERN

Orthorhombic, $P2_12_12_1$; $a=10.07$, $b=10.52$, $c=6.12$A; $Z=4$

I	d(meas.)	hkl	d(calc.)	I	d(meas.)	hkl	d(calc.)	I	d(meas.)	hkl	d(calc.)
8	7.37A	110	7.275A	9	2.83	311	2.835	2	2.26	411	2.273
10	5.34	011	5.291			320	2.830			420	2.271
		020	5.260			112	2.822			302	2.262
		101	5.231	8	2.64	022	2.645			331	2.254
2	5.04	200	5.035			040	2.630	1	2.20	312	2.211
1	4.53	210	4.542	7	2.55	321	2.568	½	2.17	241	2.178
6	3.71	121	3.709			122	2.559	½	2.13	421	2.129
½	3.53	211	3.647			140	2.545	2	2.06	340	2.070
		220	3.637			212	2.538			150	2.060
½	3.33	130	3.312	½	2.47	410	2.449	½	2.01	013	2.003
½	3.23	310	3.198	½	2.40	041	2.417			103	2.000
1	3.06	002	3.060	3	2.33	240	2.331	3	1.957	113	1.965
		031	3.043			401	2.329			341	1.961
4	2.94	301	2.943							142	1.957
		012	2.939							151	1.952
		102	2.929								

I	d(meas.)	I	d(meas.)	I	d(meas.)	I	d(meas.)	I	d(meas.)
1	1.903	½	1.537	½	1.269	½	1.090	1	0.912
1	1.859	5	1.510	½	1.237	½	1.081	1	0.900
½	1.784	2	1.455	½	1.222	1	1.054	½	0.876
½	1.746	½	1.412	1	1.207	½	1.037	½	0.843
2	1.710	½	1.389	½	1.189	1	1.026	1	0.833
2	1.664	1	1.358	½	1.163	1	0.996	½	0.822
½	1.616	1	1.341	½	1.150	1	0.974		
½	1.600	2	1.311	½	1.124	1	0.959		
1	1.570	½	1.287	½	1.104	½	0.947		

colours all give identical x-ray powder patterns. A fifth specimen (USNM C4207, St. Day, Cornwall) also gives the same pattern as evidenced by a film taken in the United States Geological Survey laboratories and kindly sent to us for comparison by Dr. Charles Milton (Feb. 11, 1948).

Crystallography. Crystals of suitable size from the first specimen referred to above were readily adjusted on the two-circle goniometer for single crystal x-ray measurements. Rotation, zero and first layer Weissenberg films about a, b and c axes yielded the following dimensions of the unit cell:

$$a=12.70, \qquad b=7.57, \qquad c=9.88 \text{ A}; \qquad \beta=91°23'$$

The β angle was determined by direct measurement on the Weissenberg film of the $(h0l)$ layer of the reciprocal lattice and by calculation from the measured spacings $d(100)$, $d(001)$, $d(101)$, $d(201)$ and $d(503)$. The above value is an average of these determinations.

The observed diffractions conform to the following extinction conditions: (hkl) present only for $h+k+l=2n$, $(h0l)$ present only for $h=2n$

and $l=2n$ and $(0kl)$ present only for $k=2n$. These extinctions are characteristic of the space group $I2/a$. The I lattice is retained because of the strong pseudo-orthorhombic habit of the crystals (Figure 12).

The cell dimensions give the axial ratio

$$a:b:c=1.678:1:1.305; \qquad \beta=91°23' \text{ (x-ray)}$$

$$c:b:a=1.6808:1:1.3191; \qquad \beta=91°27' \text{ (Dana, 1892)}$$

which compares closely to that given by Descloizeaux (Dana, 1892) when the a and c axes are interchanged. The only observed forms $m(110)$ and $e(011)$ are interchanged in transforming from Descloizeaux to the structural setting. The crystals (Fig. 12) are flattened and wedge-shaped with the forms $m(110)$ and $e(011)$ in nearly equal development; the faces of both are striated parallel to the intersection edge. Most commonly they are elongated along the new c axis with $m(110)$ larger than $e(011)$.

Composition and cell content. New determinations of the specific gravity of liroconite, 2.94 (Frondel, personal communication 1946) and 3.01 (Davies, 1946) agree closely with the old values 2.985 (Hermann, 1844), 2.926 (Haidinger) and 2.964 (Damour, 1845) given in Dana (1892).

TABLE 10. LIROCONITE: ANALYSES AND CELL CONTENT

	1	2	3	4	A	B
CuO	36.38	37.18	37.40	36.73	36.96	37.70
Al_2O_3	10.85	9.86	10.09	n.d.	10.47	12.07
Fe_2O_3	0.98	—	—	—		
As_2O_5	23.05	22.22	22.40	23.85	22.56	20.42
P_2O_5	3.73	3.49	3.24	1.02	3.49	4.20
H_2O	24.01	25.49	25.44	incomp.	25.31	25.60
	100.00	98.06	98.57	—	98.88	100.00
Cu	7.74	8.07	8.07	—	7.96	8.0
Al	3.60	3.34	3.40	—	3.51	4.0
Fe	0.20	—	—	—		
As	3.40	3.34	3.36	—	3.37	3.0
P	0.89	0.85	0.79	—	0.84	1.0
H	47.04	48.82	48.46	—	48.11	48.0
O	47.69	47.98	47.77	—	47.85	48.0

1–3. Cornwall, England, 1. anal. Hermann (1844), G=2.985. 2–3. anal. Damour (1845), G=2.964; 4. anal. Church (1895), G=2.97. Al_2O_3 not determined, H_2O incomplete. A. Average analysis and cell content from analyses 1–3. B. Ideal composition and cell content for 4 $[Cu_2Al(As, P)O_4(OH)_4 \cdot 4H_2O]$.

The average of these measured values, 2.96, combined with the cell dimensions gives the molecular weight of the unit cell M = 1693.0.

In Table 10, this value has been used to compute the atomic content of the unit cell from the three available analyses of liroconite. The average analysis and cell content (A) agree closely with the ideal figures (B) clearly indicating the cell content.

$$8CuO \cdot 2Al_2O_3 \cdot 2(As_2O_5 \cdot P_2O_5) \cdot 24H_2O = 4[Cu_2Al(As,P)O_4(OH)_4 \cdot 4H_2O]$$

for which the calculated specific gravity is 2.95 also in close agreement with the measured values. The As = P ratio of 3:1 is clearly shown by the first three analyses, the later incomplete analysis by Church (1895) indicates a higher ratio. These authors do not state the exact colour of their analysed material, therefore further analyses would be required to explain the marked difference in colour between different specimens.

TABLE 11. LIROCONITE—$Cu_2Al(As,P)O_4(OH)_4 \cdot 4H_2O$: *X*-RAY POWDER PATTERN

Monoclinic, *I*2/*a*; $a = 12.70$, $b = 7.57$, $c = 9.88$A, $\beta = 91°23'$, $Z = 4$

I	*d*(meas.)	*hkl*	*d*(calc.)
10	6.46A	110	6.502A
9	5.95	011	6.008
1	4.33	211	4.332
5	3.92	$\bar{1}12$	3.957
		112	3.910
1	3.66	310	3.694
2	3.36	121	3.398
10	3.01	013	3.013
		022	3.004
		$\bar{3}12$	2.988
½	2.91	312	2.929
5	2.79	$\bar{4}11$	2.824
		411	2.790
6	2.69	321	2.701
		222	2.700
		$\bar{4}02$	2.700
½	2.63A	402	2.641A
3	2.47	130	2.475
		004	2.469
4	2.44	$\bar{1}23$	2.446
		420	2.432
3	2.27	204	2.283
		231	2.277
5	2.21	$\bar{1}32$	2.217
		$\bar{4}22$	2.198
		$\bar{5}12$	2.184
5	2.15	330	2.167
		422	2.166
		$\bar{3}23$	2.159
		512	2.145
3	2.07A	$\bar{3}14$	2.073A
		$\bar{5}21$	2.070
½	1.994	033	2.003
		$\bar{3}32$	1.994
2	1.914	$\bar{2}33$	1.918
		015	1.911
½	1.834	141	1.838
1	1.787	$\bar{5}23$	1.792
		530	1.790
4	1.749	710	1.764
		$\bar{1}34$	1.752
		$\bar{6}22$	1.742
4	1.707	$\bar{2}42$	1.706
		$\bar{3}41$	1.705

I	*d*(meas.)
1b	1.629A
1b	1.588
3b	1.544
3b	1.512
2	1.457
1	1.410
1	1.388A
1	1.366
1	1.342
½	1.320
½	1.282
½	1.254
1	1.216A
½	1.198
½	1.174
½	1.121
½	1.107
½	1.080
½	1.058A
½b	1.035
½b	1.016
½b	0.988
½	0.949
1	0.938
½	0.922
½	0.909
½	0.860
½	0.852
½b	0.830
½b	0.811

b—broad line.

X-ray powder pattern. The *x*-ray powder pattern of liroconite (CuK radiation, Ni filter) is shown in Figure 8. The observed relative intensities and measured spacings of the diffraction rings are listed in Table 11 together with indices and interplanar spacings calculated from the lattice dimensions down to $d = 1.707$A.

OLIVENITE AND "LEUCOCHALCITE"

Olivenite is the most commonly observed arsenate of copper found in nature. Lattice dimensions and space group data for olivenite are given by Heritsch (1937) and Richmond (1940). The lattice constants given by these authors are in reasonable agreement but the space groups differ markedly.

Materials. Several typical specimens of olivenite were available for study in Queen's University mineral collections and other specimens appeared during the examination of specimens labelled erinite, cornwallite and conichalcite. The principal localities for these specimens are Cornwall and Tintic, Utah.

Specimens of leucochalcite from Wilhelmine Mine (type locality) and Sommerkahl mine, Schollkrippen, Spessart, Germany were available from Harvard University mineral collections. Several specimens showing olivenite and leucochalcite from Majuba Hill, Nevada were kindly provided for study by Hatfield Goudey. Leucochalcite on the German specimens consists of small groups of radiating or matted silky fibres of greyish colour on a fracture surface in gneiss. Leucochalcite from Nevada is more abundant on our specimens. On one specimen it consists of matted silky fibres in openings in much altered rhyolite heavily stained with limonite. A second specimen shows a silky fibrous coating with a slight greenish colour with tyrolite on a fracture surface. A third specimen consists of fine silky matted and radiating fibres with a delicate greenish white colour. A fourth specimen shows solid crystals of typical olivenite grading into fine silky greenish white fibres in a rock cavity. All the above specimens of leucochalcite gave an *x*-ray powder pattern identical with that of olivenite (Fig. 9, 10).

X-ray studies. A typical crystal of olivenite was mounted on the two-circle optical goniometer and adjusted to rotate about the *c* axis. Rotation, zero, and first layer Weissenberg films were taken of this crystal about the *c* axis. Many of the diffractions ($h0l$) and ($0kl$) were so weak as to render the space group characteristics difficult to determine. Therefore zero layer films were taken about the *a* and *b* axes on the Precession camera. These films were taken on the same crystal without remounting.

The films yielded the lattice dimensions shown below in comparison with those of Heritsch (1937) from rotation and powder films and Richmond (1940) from rotation and Weissenberg films. A fibre of leucochalcite gave a weak rotation film with the *c* length of olivenite.

$a=8.22$, $b=8.64$, $c=5.95$ A (Heritsch, 1937)[1]

$a=8.18$, $b=8.56$, $c=5.87$ A (Richmond, 1940)[1]

$a=8.24$, $b=8.64$, $c=5.95$ A (olivenite, L.G.B.)
$c=5.96$ A ("Leucochalcite," L.G.B.)

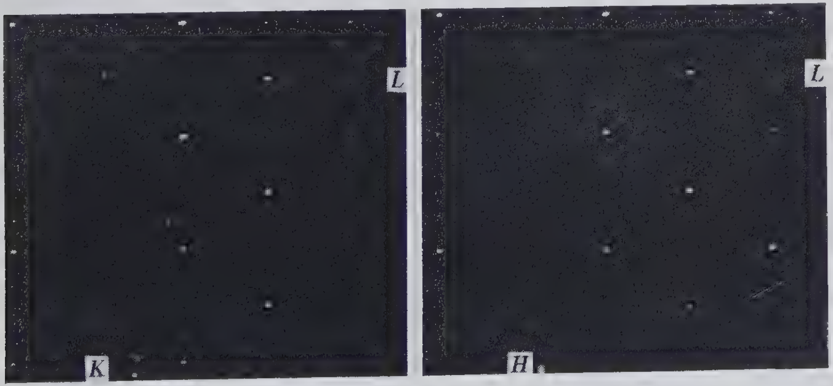
From indexing the equator layer line on rotation films Heritsch concluded that the space group should be D_{2h}^{1} not D_{2h}^{12} as in libethenite (Strunz, 1936). Richmond (1940) determined the space group as D_2^4 from Weissenberg films about a and c. The films obtained in the present study, particularly the $(0kl)$ and $(h0l)$ layers on precession films (Figs. 13, 14) also the $(hk0)$ layer on a Weissenberg film clearly indicate the following extinction conditions: (hkl) all present; $(0kl)$ all present; $(h0l)$ present only with $h+l=2n$; $(hk0)$ all present. These criteria agree with those observed by Heritsch and are characteristic of the space group $D_{2h}^{13}-Pmnm$ if the class is holohedral. It is probable that D_{2h}^{1} is a misprint for D_{2h}^{13} in Heritsch's paper.

Chemical composition. The analyses combined with the cell dimensions and measured specific gravity clearly show the following cell content for olivenite:

$$Cu_8As_4H_4O_{20}=4[Cu_2AsO_4(OH)]$$

The calculated specific gravity for this cell content and Heritsch's lattice dimensions is 4.45 in close agreement with the measured value 4.378.

X-ray powder pattern. The intensities and interplanar spacings for the diffractions observed on the x-ray powder films of olivenite and leucochal-



FIGS. 13, 14.—Olivenite, zero layer Precession films, Cu/Ni radiation, film to specimen distance 60 mm. reproduced 2/3 actual size. Fig. 13. $(0kl)$ showing weak diffractions (012), (023); very weak diffractions (021), (032), (041) are also present on the film. Fig. 14. $(h0l)$ showing diffractions present with $h+l=2n$ only.

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[1] Lattice dimensions converted to new Angstrom units (1946).

cite are given in Table 12. The pattern has been indexed to $d = 1.488$ A with close agreement between measured and calculated spacings.

TABLE 12. OLIVENTE—$Cu_2AsO_4(OH)$: *X*-RAY POWDER PATTERN
Orthorhombic, *Pmnm*; $a = 8.22$, $b = 8.64$, $c = 5.95$A, $Z = 4$

I	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)
7	5.91A	110	5.955A	7	2.39A	202	2.410A	1	1.740A	213	1.749A
9	4.82	011	4.900			311	2.391			042	1.748
		101	4.820	1	2.33	122	2.348	1	1.686	402	1.691
6	4.19	111	4.209			212	2.321			150	1.691
4	3.80	120	3.824	½	2.16	040	2.160	2	1.647	332	1.651
10	2.98	220	2.977	½	2.08	140	2.089			223	1.651
		002	2.975			032	2.069	1	1.621	151	1.627
4	2.70	130	2.718	1	1.973	330	1.985			510	1.615
6	2.65	221	2.663			141	1.971	3	1.600	242	1.608
		112	2.661			312	1.963			303	1.607
1	2.59	310	2.612	½	1.925	013	1.933			133	1.602
		031	2.592			103	1.928	6	1.575	313	1.580
7	2.47	301	2.489	2	1.881	411	1.895			422	1.574
		131	2.472			331	1.883	½	1.556	511	1.559
		022	2.450			113	1.882	5	1.488	440	1.489
				½	1.831	322	1.826			004	1.487

I	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)
½	1.439A	3	1.290A	1	1.106A	2	1.002A	2	0.895A
½	1.419	1	1.281	½	1.096	2	0.984	1	0.881
1	1.404	2	1.252	1	1.090	2	0.973	3	0.872
½	1.386	½	1.192	2	1.079	½	0.964	1	0.862
1	1.364	½	1.177	½	1.068	½	0.959	½	0.856
2	1.354	1	1.166	4	1.052	½	0.949	2	0.843
4	1.327	½	1.126	1	1.031	½	0.915	2	0.816
1	1.305	½	1.114	1	1.015	½	0.904		

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NOTES AND NEWS

MINERAL OCCURRENCES IN WESTERN CANADA

R. M. Thompson, *University of British Columbia, Vancouver, B. C.*

These notes continue the series, the last of which appeared in *Am. Mineral.* **35,** 451, 1950. All identifications were established or confirmed by *x*-ray powder photographs. The writer wishes to acknowledge the helpful assistance of Dr. H. V. Warren and the following students who cooperated by collecting and/or examining ores: R. J. Charles, E. Livingston, G. A. Noel, C. Rennie, R. A. Stuart, and A. Sutherland-Brown.

Berthierite. $FeS \cdot Sb_2S_3$. Clearwater Lake, Alice Arm, Portland Canal M.D., B.C. This lake lies at the head of the Kitsault River about 40 miles north of Alice Arm. Specimens collected by R. W. Goranson but not closely identified with respect to prospect or locality, consist of a 1 inch quartz vein with slender needles of berthierite ("stibnite") with a marked iridescent tarnish lying perpendicular to the walls. This is believed to be the first recognized occurrence of this mineral in British Columbia.

Boulangerite. $5PbS \cdot 2Sb_2S_3$. Silver Key Group, near Canal Flats, Golden M.D., B.C. This property also known as the Key Group is situated at the head of the east fork of Doctor Creek about 25 miles west of Canal Flats (*Minist. Mines, B.C., Ann. Rept.*, 1935, p. E 28). Lenses and wedges of sulphides occurring along the bedding planes of highly metamorphosed, sericitized, and contorted quartzite consist of galena, pyrite, and minor amounts of chalcopyrite, sphalerite, and possibly an antimonial sulphide.

Samples provided by Dr. P. H. Sevensma and Mr. A. C. Taplin, vary from massive gneissic galena intimately mixed with boulangerite to mixtures of quartzite with siderite containing needles of and vugs lined with boulangerite. Assays of over 200 oz. of silver per ton are believed to be due to argentiferous tetrahedrite but none was seen in the samples examined.

Mastadon Property, Revelstoke M.D., B.C. Meneghenite and associated minerals have already been described from this property (Warren, *Univ. Toronto Studies*, Geol. Ser., **51,** 75, 1947). A recent hand specimen supplied by Dr. D. F. Kidd and thought to contain meneghenite consists of a narrow band of fibrous boulangerite with galena and sphalerite adjacent to a mass of quartz and sericite.

Braunite. $(Mn, Si)_2O_3$. Iron King, near Olalla, Osoyoos M.D., B.C. (*Minist. Mines, B.C., Ann. Rept.*, 1949, 132). This prospect is on the south side of the ridge separating the north and middle forks of Olalla

Creek, about $3\frac{1}{2}$ miles by trail from the highway at Olalla, and at elevations from 3,800 to 5,300 feet. Here a bedded deposit of manganiferous chert is traversed by a network of minute veins containing rhodonite, which merge laterally into zones of hard black siliceous manganese ore (braunite) containing small irregular masses of rhodonite and chert.

Specimens supplied by Dr. W. H. White and Mr. Kennedy show massive pinkish chert traversed by thin streaks of braunite and rhodonite while others vary from a mixture of quartz, rhodonite, rhodochrosite, hematite, and braunite, to almost solid braunite. The braunite appears to be an alteration product of the rhodonite and is cut by many tiny stringers of rhodochrosite and quartz. This mineral has not been recorded previously in British Columbia.

Cobaltite. CoAsS. Laverdiere Group, Atlin M.D., Atlin, B.C. This property is situated about 2 miles from Atlin Lake on the west side of Hoboe Creek at the contact of early Paleozoic amphibolites, schists and limestone and a granitic intrusive which is part of the Coast Range Batholith (Cairnes, *Geol. Surv. Canada, Mem.* **37,** 117, 1913).

Samples collected by Mr. E. Livingston are mainly greenish-black serpentine with a heavy erythrite coating. Fresh surfaces show disseminated or massive granular magnetite, and minor amounts of chalcopyrite, specularite, pyrite, and cobaltite which was apparently overlooked in the earlier work.

Hedleyite. Bi_7Te_3. Oregon Property, near Hedley, Osoyoos M.D., B.C. This property is situated between Sixteen Mile and Eighteen Mile Creeks about 3 miles east of Hedley. The mineralization here is similar to that of the Good Hope Claim (Warren and Peacock, *Univ. Toronto Studies*, Geol. Ser., **49,** p. 55–69, 1945), about 2 miles north, and consists of massive garnet, hedenbergite, wollastonite, calcite, and minor quartz with sparsely disseminated sulphides.

Hedleyite and Joseite B are seen together in polished sections as rounded grains with smooth boundaries. When the two minerals are in contact, hedleyite is distinguished by its slightly lighter colour. Native bismuth, molybdenite, and gold are closely associated with the tellurides while bornite, chalcopyrite, cobaltite, and safflorite occur apart and are locally abundant.

Meneghenite. $Cu_2S \cdot 26PbS \cdot 7Sb_2S_3$. Silver Bell Claim of the California Group, Chikamin Mountain, Whitesail Lake, Omineca M.D., B.C. This property is situated on the south facing slope at the west end of Chikamin Mountain at an elevation of 6,500 feet. Samples collected from the dump consist of limonite stained granular to vuggy quartz and minor siderite with bands of galena, yellow sphalerite, tetrahedrite, and silvery gray needles and prisms of meneghenite ("stibnite") up to 1 cm. in length. Polished sections show small rounded areas of bournonite in contact with tetrahedrite, and associated with galena, sphalerite, and chalcopyrite, while the meneghenite laths are usually free.

Durango (Howard) Mine, near Ymir, Nelson M.D., B.C. This property is located on the east side of Active (South Fork of Porcupine) Creek, about 9 miles south-east of Ymir. Investigation of a specimen of steely galena kindly supplied by the B.C. Department of Mines showed blebs of chalcopyrite, lenses of covellite and a few beaded stringers of a gray anisotropic mineral which proved to be meneghenite.

Silver Standard Mine, near New Hazelton, Omineca M.D., B.C. This mine is situated about 6 miles by road northwest of New Hazelton on the west side of Glen Mountain between 1,200 and 2,000 feet elevation. Meneghenite was discovered during a detailed mineralographic examination of this ore. It occurs in close association with argentiferous tetrahedrite, galena, pyrargyrite, and polybasite as fine laths and fibrous masses. It is less white than galena and shows prominent cleavage along which alteration to anglesite is taking place. Traces of bournonite are present as isolated blebs associated with the meneghenite which association has been noted in three of the four occurrences of meneghenite in British Columbia. Sphalerite, pyrite, pyrrhotite, marcasite, arsenopyrite, chalcopyrite, covellite, and limonite are also present.

Parkerite. $Ni_3Bi_2S_2$. Near Gros Cap, N.W.T. This rare mineral was reported by Stockwell (*Geol. Surv. Canada*, Summ. Rept., 1932, Part C, pp. 60–61) as an unknown soft bluish-gray anisotropic mineral in niccolite from a prospect containing nickel and cobalt-bearing minerals at a point 2 miles north of Great Slave Lake and 3 miles east of Francois River, N.W.T. Specimens collected from this prospect by Mr. G. A. Dirom in 1932 were recently examined by the writer and found to consist of closely spaced compact masses of botryoidal niccolite and rammelsbergite cemented by a carbonate gangue and covered with much earthy annabergite. A small amount of native bismuth was noted in gangue at one corner of a specimen.

Polished sections show rounded areas of niccolite often bordered by a narrow band of rammelsbergite and sometimes areas of niccolite containing irregular to rounded areas of rammelsbergite. In one section, laths of niccolite are seen radiating from the centre of crudely star-shaped areas of rammelsbergite. The more massive areas of niccolite carry a series of bluish-gray dots and blebs of parkerite (0.01 mm.) along an arc parallel to its outer botryoidal surface. Rarely, somewhat larger areas of parkerite (0.02 mm.) are seen with distinct multiple twinning. The quantities of parkerite present however were insufficient to determine the position of this mineral in the parkerite ($Ni_3Bi_2S_2$)-shandite ($Ni_3Pb_2S_2$) series (Peacock and McAndrew, *Am. Mineral.*, **35,** 425–439, 1950).

Polybasite. $8(Ag,Cu)_2S \cdot (Sb,As)_2S_3$. Torbrit Silver Mines Limited, Portland Canal M.D., B.C. The Torbrit (Toric) Mine is loacted on the east bank of the Kitsault River, 17 miles by road from Alice Arm. Samples examined were taken from a zone of high grade silver ore and consist of a mixture of calcite, barite, jasper, hematite, quartz, and altered greenstone fragments impregnated with grains of pyrite, galena, irregular patches of fine grained ruby silver, and a minor amount of native silver. The overall effect is a mottling but some specimens show a crude banding.

Polished sections show pyrargyrite as disseminated grains and as 1 mm. veinlets with polybasite. Complications in milling may be expected due to the fineness and brittle nature of the silver minerals.

Rammelsbergite. $NiAs_2$. Nix Property, Great Slave Lake, N.W.T. This property is located about 10 miles S.W. of the Taltheilei Narrows in the N.E. arm of Great Slave Lake and is approximately 50 miles northeasterly from the Gros Cap property described above.

Specimens kindly supplied by Dr. H. C. Gunning (which are almost identical to those from the Gros Cap) consist of solid compact masses of niccolite and rammelsbergite in a minor amount of ankeritic gangue and partially coated with anabergite.

Polished sections show small botryoidal areas of niccolite bordered by narrow bands of rammelsbergite; also somewhat feathery areas of niccolite in a groundmass of rammelsbergite. No parkerite was observed in these specimens.

Stephanite. $5Ag_2S \cdot Sb_2S_3$. Elsa Mine, near Mayo, Y.T. This mine is located on the north facing slope of Galena Hill, 27 miles north of Mayo. A specimen kindly supplied by Mr. H. Gabrielse from the 200 foot level consists of a narrow limonite stained $\frac{1}{4}$ inch quartz vein sparsely mineralized with pyrite and covered on one side with small flat areas of massive black stephanite suggestive of a secondary origin. This mine is well known for its high silver values which are contained in freibergite, galena (with myriads of dots and blebs of unidentifiable ruby silvers), argentite, and native silver. The first occurrence of this rare silver sulphosalt in recognizable quantities in Western Canada is of interest.

Tellurbismuth. Bi_2Te_3. Fil-Mil Mine, Deep Inlet, Alberni M.D., B.C. This property is on the south side of Deep Inlet, about 2 miles from its entrance into Kyuquot Sound and 15 miles by boat from Kyoquot (*Minst. Mines, B.C., Ann. Rept.*, 1947, 177). A number of narrow fissures and shear zones in granodiorite are filled with quartz and aplitic material and sparsely mineralized with pyrite, sphalerite, chalcopyrite, and gold. A small sample supplied by the B.C. Chamber of Mines shows a coarse aggregate of quartz crystals cemented by gold and plates of tellurbismuth. Bismuth tellurides are becoming common accessory minerals in a number of gold occurrences in this province.

Tetrahedrite-Tennantite. $(Cu, Fe)_{12}(Sb,As)_4S_{13}$. Taylor Windfall Mine, Battlement Creek, Clinton M.D., B.C. This property is on Battlement Creek adjoining its junction with the Taseko River about 9 miles easterly from Taseko Lake (*Minist. Mines B.C., Ann. Rept.*, 1935, F 19). Included in a number of specimens kindly supplied by Mr. R. H. Stewart is a 6×4×3 inch solid compact mass of silvery-gray sericite which contains disseminated crystals and masses of tetrahedrite and pyrite. The tetrahedrite shows poor to fairly well developed tetrahedral crystals up to ¼ inch in size. A number of crystals were carefully selected and specific gravity measurements made on each. A chemical analysis was made by Mr. R. N. Williams on a sample with an average specific gravity of 4.68. A polished section of a portion of the material used for analysis showed only tetrahedrite while a section of fragments with a specific gravity below 4.68 showed chalcopyrite as the main impurity. This analysis (A) is given below together with the values calculated to 100 per cent (B).

	A	B
Cu	35.03	35.12
Fe	2.65	2.66
Zn	8.50	8.52
Pb	2.05	2.06
Sb	12.54	12.57
As	12.48	12.51
S	26.49	26.56
	99.75	100.00

This analysis would tend to confirm the suggestion in Dana (1944, p. 375) that Sb-As apparently form a complete series from tetrahedrite to tennantite, respectively, with the above mineral occupying the mid position in this series.

Uraninite. UO_2. Emerald Mine, near Salmo, Nelson M.D., B.C. The Emerald property is located 6 miles south-east of Salmo and extends across the height of land between Sheep and Lost Creeks. During the last war this mine became an important producer of tungsten and for a full account the reader is referred to *B.C. Department of Mines*, Bull. **10,** p. 135, 1943.

In the spring of 1948 the Metallurgy Department of this University requested the writer to identify a fine black powder which came off a

Wilfley Table ahead of the scheelite from this mine. A binocular inspection showed a black finely crystalline powder which was later determined to be uraninite at the University of Toronto. On receipt of this information the company made a thorough search of the mine and provided a suite of ore which was considered to be the most likely for the occurrence of radioactive minerals. This material which consists largely of quartz, pyrrhotite, pyrite, chalcopyrite, and minor molybdenite, failed unfortunately to produce further amounts of uraninite. From the above it would appear that the uraninite was a local accessory mineral but its occurrence warrants a close watch on further developments at this mine and in the surrounding area.

THE CHILDRENITE-EOSPHORITE PROBLEM

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On the basis of an exhaustive optical study of the childrenite-eosphorite series, Professor C. S. Hurlbut (*Am. Mineral.*, **35,** 793–805, 1950) concludes that these minerals should be classified in the monoclinic system. Previous morphological (*Dana's Textbook of Mineralogy*, 732, 1932) and x-ray diffraction (Barnes, *Am. Mineral.*, **34,** 12–18, 1949) examinations have placed childrenite in the orthorhombic system.

Professor Hurlbut has found the same x-ray extinctions for a single and for a twinned crystal of Newry eosphorite as those previously reported by one of us (Barnes, *Am. Mineral.*, **34,** 12–18, 1949) for Tavistock childrenite. Through the kindness of Professor Hurlbut we have been able to examine crystals of his Newry eosphorite optically and have confirmed the twinning and extinction angle. We have also observed his fourlings in a crystal of childrenite ground parallel to (010). There is thus no conflict regarding the experimental data which, on the basis of x-ray diffraction symmetry alone, leads to the space group $Bba2$ or $Bbam$ but apparently must be reduced to $B2_1/a$ (with $\beta = 90°$) to accommodate the optical data.

The essential difference between the diffraction symmetry of the monoclinic space group $B2_1/a$ (with $\beta = 90°$) and that of the orthorhombic space group $Bba2$ (or, $Bbam$) is the b glide plane perpendicular to a in the latter. This can readily be observed by comparing the reciprocal lattice nets for the zero and upper levels of crystals belonging to the two space groups (see, for example, Figure 5 of a previous paper (Barnes, *Am. Mineral.*, **34,** 166, 1949) on lindgrenite $B2_1/c$ where the c glide is equivalent to an a glide and β may be visualized as 90°, and Figure 7 of a previous paper (Barnes, *Am. Mineral.*, **34,** 16, 1949) on childrenite $Bba2$).

Thus, although the *b* glide in *Bba*2 automatically introduces 2_1 along *b*, a *b* glide is not introduced in $B2_1/a$ by 2_1. In the figures mentioned it will be observed that the first levels of the a^*c^* nets, the second levels of the a^*b^* nets and the zero levels of the b^*c^* nets are not identical. In terms of extinctions, (0*kl*) reflections should be absent when $(k+l)$ is odd in *Bba*2 due to the *b* glide, but should be present in $B2_1/a$ except in the special case (0*k*0).

In order to be certain that possible weak reflections forbidden by *Bba*2 had not been missed in earlier work, precession photographs have been taken of crystals of Devonshire and of Cornwall childrenite and of Rumford (Black Mountain), Buckfield and Newry eosphorite. As a final test, zero level, *a* axis Weissenberg photographs of a Rumford eosphorite crystal were obtained with *Mo* radiation and various exposure times. Results are shown in Table 1.

TABLE 1

O level, *a* axis Weissenberg, *Mo* radiation

Exposure (hours)	Number of reflections		
	Observed	Possible	
		$B2_1/a$	*Bba*(2)
5	33	102	54
20	63	218	118
100	91	408	210

By "possible" reflections is meant the total number permitted by the space group over the *sin θ* range for which spots appeared on the photographs. As Table 1 shows, there were 48 more reflections possible for $B2_1/a$ than for *Bba*2 on the first film, 100 on the second and 198 on the third film. *Not one of these spots was present.* It seems most unlikely that every one was absent because its intensity was too weak to affect the film in the times of exposure employed.

There is no doubt, therefore, that a real anomaly exists between the optical and *x*-ray diffraction data in the case of the childrenite-eosphorite series. We have started a systematic structure investigation in the hope that the answer is to be found there.

In this connection we have re-examined the possible piezoelectric properties of various childrenite and eosphorite crystals using a detector modelled after that of Stokes (*Am. Mineral.*, **32,** 670–677, 1947). We have been unable to get a positive effect so that it is possible that childrenite and eosphorite, if orthorhombic, should be assigned to the space group *Bbam* rather than to *Bba*2. Both space groups are being considered in the structure investigation.

THE UNIT CELL OF LINARITE

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Linarite, a basic sulphate of lead and copper, occurs in deep azure blue monoclinic crystals. The colour is distinctly brighter than azurite. The crystals are elongated along b and have been described as tabular on $c(001)$ also on $s(\bar{1}01)$. The opportunity to make a single crystal x-ray study of this mineral was provided by a specimen of linarite with cerussite from Red Gill, Cumberland from the mineral collections of Queen's University. Two other specimens yielded material for this study, one labelled "mixite" (Queen's Museum) from American Eagle Mine, Tintic, Utah, and the other from an unknown locality. Much of the formal work was done as student exercises in a graduate course at Queen's University.

Rotation, Weissenberg and later Precession films yield the following crystal lattice dimensions expressed in terms of two closely similar unit cells.

I	$a=9.70$	$b=5.65$	$c=4.68$ A,	$\beta=102°40'$
II	$a=9.80$	$b=5.65$	$c=4.68$ A,	$\beta=105°04'$

The systematic extinctions, $(0k0)$ present only with $k=2n$, are characteristic of the space group $P2_1/m$ in either setting. Morphological studies indicate holohedral symmetry for linarite.

The unit cell dimensions give axial ratios in close agreement, I with Kokscharov (1869, Dana, *System*, 1892) and II with Goldschmidt (*Winkeltabellen*, 1897).

I	$a:b:c=1.7168:1:0.8283$	$\beta=102°40'$
	$a:b:c=1.71613:1:0.82962$	$\beta=102°37\frac{1}{2}'$ (Kokscharov)
	$a:b:c=1.7216:1:0.8297$	$\beta=102°41'$ (Robertson, 1925, *Min. Abs.* **3,** 295, 1928)
II	$a:b:c=1.7346:1:0.8283$	$\beta=105°04'$
	$a:b:c=1.7352:1:0.8296$	$\beta=105°11'$ (Goldschmidt)

The structural setting (I), which has the least oblique cell is the setting chosen by Kokscharov (1869) and followed by Dana (1892). Our crystals all show flattening on $s(\bar{1}01)$. In Goldschmidt's setting the plane of flattening becomes $c(001)$. The settings are related by the transformation formula $\bar{1}0\bar{1}/010/001$ (Kokscharov to Goldschmidt).

Table 1 gives the x-ray powder pattern for linarite indexed as far as the spacing 2.09 A. The measured spacings are in substantial agreement with the data given by Waldo (*Am. Mineral.*, **20,** 575, 1935). The pattern is reproduced in Figure 1.

New determinations of the specific gravity of linarite

5.30	Red Gill, Cumberland	(Berman balance)
5.25	Mono County, California	C. Frondel, private communication—May 13, 1946
5.26	Rezbanya, Hungary	

combined with the volume of the unit cell and any of the closely agreeing analyses clearly indicate a structural formula:

$$2PbO \cdot 2CuO \cdot 2SO_3 \cdot 2H_2O = 2[PbCuSO_4(OH)_2]$$

with calculated specific gravity 5.319.

TABLE 1. LINARITE—$PbCuSO_4(OH)_2$: *X*-RAY POWDER PATTERN
Monoclinic, $P2_1/m$; $a=9.70$, $b=5.65$, $c=4.68$A, $\beta=102°40'$; $Z=2$

I	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)	*I*	*d*(meas.)	*hkl*	*d*(calc.)
2	4.82A	110	4.851A	1	2.81A	020	2.825A	4	2.16A	410	2.182A
4	4.48	$\bar{1}01$	4.518	1	2.68	120	2.707			311	2.181
7	3.53	210	3.628	3	2.56	$\bar{3}11$	2.587			$\bar{1}12$	2.161
		011	3.551	1	2.39	021	2.402	4	2.09	320	2.105
10	3.12	300	3.155			$\bar{1}21$	2.395			$\bar{2}02$	2.097
		111	3.153	3	2.30	$\bar{4}01$	2.318			$\bar{3}02$	2.078
		$\bar{2}11$	3.105	3	2.24	$\bar{2}02$	2.259				
2	2.94	201	2.977			$\bar{2}21$	2.249				

I	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)	*I*	*d*(meas.)
6b	1.791A	½	1.532A	1	1.324A	½	1.118A	1b	0.869A
1	1.758	½	1.514	½	1.299	2	1.056	1b	0.860
½	1.734	1	1.492	½	1.287	1	1.047	1b	0.849
2	1.675	½	1.439	1	1.214	½	1.025	2b	0.839
½	1.631	1	1.400	2	1.192	1	0.993	½b	0.830
½	1.610	1	1.375	1	1.152	1	0.914	1	0.821
3	1.566	½	1.347	½	1.130	1b	0.896	1	0.813

b—broad line; using CuKα=1.5418A, mass factor 1.6602.

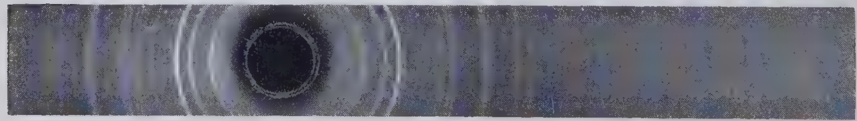

FIG. 1.—*X*-ray powder photograph, Cu/Ni radiation, 1°θ=1 mm. on film, actual size print.

THE UNIT CELL OF MAGNETOPLUMBITE

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Magnetoplumbite was described by Aminoff (*Geol. För. Förh.* **47,** 283, 1925) from Långban, Sweden, with cell dimensions and analysis by Almström. The mineral was later re-analysed by Blix (*Geol. För. Förh.* **59,** 300, 1937); this later analysis showing the true valence state of the manganese. Adelsköld (*Ark. Kemi.*, **12** (29), 1938) determined lattice dimensions for $PbO \cdot 6Fe_2O_3$ which he states corresponds to magnetoplumbite. In Dana (*System*, **1**, 728, 1944) it is shown that the structural formula deduced from the mineral data bears no similarity to the composition of the artificial lead ferrite.

Recently a powder photograph (Figure 1, Fe/Mn radiation) was taken of magnetoplumbite from a specimen from the type locality (Queen's University Museum). The pattern could not be indexed with the cell dimensions of Aminoff. A small fragment was detached and oriented on the two-circle goniometer with the perfect basal cleavage (0001) polar to the axis of the vertical circle. Rotation, zero and first layer Weissenberg films about the *c*-axis yielded the following cell dimensions

$a=5.88$, $c=23.02$A (Magnetoplumbite)[1]

$a=6.06$, $c=23.69$A (Aminoff, 1925)

$a=5.877$, $c=23.02$A ($PbO \cdot 6Fe_2O_3$, Adelsköld, 1938)

The systematic extinctions, $(h.h.\overline{2h}.l)$ present only with $l=2n$ are characteristic for the space group $C6/mmc$ as found by Adelsköld for artificial $PbO \cdot 6Fe_2O_3$.

The new cell dimensions combined with the measured specific gravity, 5.517 (Dana) give the molecular weight of the cell contents M = 2290.6. The cell content is calculated from the analysis by Blix in Table 1.

TABLE 1. MAGNETOPLUMBITE: ANALYSIS AND CELL CONTENT

	1	2		3		4	
PbO	20.02	2.08	Pb	2.08	2.08	2	2
MnO	3.73	1.21	Mn''	1.21	23.37		24
Al_2O_3	1.86	0.41	Al	0.82		1	
Fe_2O_3	52.22	7.55	Fe'''	15.10		15	
Mn_2O_3	17.27	2.52	Mn'''	5.04		7	
TiO_2	4.14	1.20	Ti	1.20		1	
Rem	0.76		O	37.13	37.13		38
	100.00						

1. Magnetoplumbite, Långban, Sweden, anal. Blix (1937). 2. Unit cell content. 3. Atomic content of unit cell. 4. Ideal content corresponding to $Pb_2(Fe_{15}Mn_7Al,Ti)O_{38}$.

The number of atoms (3) indicates a structural formula $Pb_2(Fe_{15}Mn_7AlTi)O_{38}$ with calculated specific gravity 5.59 in close agreement with the measured value. This structural formula is the same as for the arti-

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[1] Using wavelength CrKα 2.2909A, Mass factor 1.6602.

TABLE 2. MAGNETOPLUMBITE—$Pb(Fe,Mn,Al,Ti)_{12}O_{19}$: *X*-RAY POWDER PATTERN
Hexagonal, *C6/mmc*: $a=5.88$, $c=23.02$A, $Z=2$

I	θ(Fe)	*d*(meas.)	*hkīl*	*d*(calc.)	*I*	θ(Fe)	*d*(meas.)	*hkīl*	*d*(calc.)
1	11.2°	4.99A	$10\bar{1}1$	4.972A	3	19.7°	2.87A	0008 $11\bar{2}2$	2.878A 2.849
1	11.95	4.68	$10\bar{1}2$	4.657					
½	13.15	4.26	$10\bar{1}3$	4.243	10	20.5	2.77	$10\bar{1}7$	2.763
2	14.6	3.84	0006 $10\bar{1}4$	3.837 3.814	10	21.6	2.63	$11\bar{2}4$	2.618
					½	22.3	2.55	$20\bar{2}0$	2.546
½	16.45	3.42	$10\bar{1}5$	3.415	5	23.5	2.43	$20\bar{2}3$	2.417
5	19.1	2.96	$11\bar{2}0$	2.940	4	25.65	2.24	$20\bar{2}5$	2.228
					3	27.05	2.13	$20\bar{2}6$	2.122

I	θ(Fe)	*d*(meas.)	*I*	θ(Fe)	*d*(meas.)	*I*	θ(Fe)	*d*(meas.)
1	29.95°	1.940A	7	36.7°	1.621A	½	59.95°	1.119A
1	32.35	1.810	6	40.95	1.478	1	60.95	1.108
½	34.5	1.710	2	44.35	1.386	3	61.75	1.100
5	35.4	1.672	½	47.35	1.317	2	62.4	1.093
1	36.0	1.648	1	47.95	1.304	2	70.95	1.025
2	36.3	1.636	2	55.45	1.176	1	73.3	1.011

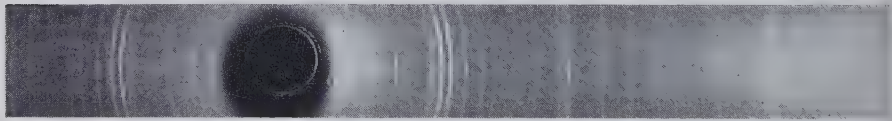

FIG. 1.—*X*-ray powder photograph, Fe/Mn radiation, 1°θ=1 mm. on film, actual size print.

ficial ferrite $2[PbFe_{12}O_{19}]$ given by Adelsköld, with substitution of manganese, aluminum and titanium for iron.

The estimated intensities and measured interplanar spacings from the powder pattern (Fig. 1) are given in Table 2 together with indices and spacings calculated from the cell dimensions.

WALKER MINERALOGICAL CLUB*

OFFICERS 1950

Honorary President, A. L. Parsons, *President,* M. H. Frohberg, *Past President,* G. E. Steel, *Secretary-Treasurer,* W. M. Tovell, *Editor,* M. A. Peacock, *Councillors,* F. Ebbutt, G. G. Waite, W. F. Take, V. B. Meen.

ABSTRACT OF PROCEEDINGS, 1950

February 16, 1950. Mr. Edward B. Tiffany, Certified Gemologist, Henry Birks and Sons Limited, Toronto, spoke to the Club on "A Trip to the Diamond Mines of South Africa." The talk was illustrated with colour slides.

April 20, 1950. Professor Harry V. Warren, Department of Geology and Geography, University of British Columbia, addressed the Club on the subject "Biogeochemical and Other Researches at the University of British Columbia." Dr. Warren discussed the mineral content of plants in relation to the mineral deposits over which they grow.

Professor George Brownell, Head of the Department of Geology at University of Manitoba, was present and described the development of the scintillometer, a new device for the detection of radioactivity from the air.

September 29–October 1, 1950. On the occasion of the Fourth Annual Field Trip, the Club met at Gouverner, St. Lawrence County, New York. Mr. D. E. Jensen, Head of the Mineral Department of Ward's Natural Science Establishment, Rochester, N. Y., and Dr. B. M. Shaub, Department of Geology, Smith College, Northampton, Mass., two Club members, acted as leaders. The high point of the collecting was at the Pierrepont locality where excellent black tourmaline crystals were obtained. Other localities visited were the McLear Pegmatite, Balmat Zinc District and Loomis Talc deposits. The party, consisting of members and their families, totalled about seventy.

December 14, 1950. Dr. S. C. Robinson, Geological Survey of Canada, addressed the Club on "Mineralogy of the Goldfields Area, Saskatchewan." Dr. Robinson gave a complete geological and mineralogical description of this area which is interesting for the association of many radioactive minerals.

At this meeting tribute was paid to the late Dr. Martin A. Peacock, under whose editorship "Contributions to Canadian Mineralogy" gained wide recognition.

It was announced that henceforth the Walker Mineralogical Club Prize would be known as the Peacock Memorial Prize. The prize for 1950 was awarded to R. J. Arnott for his paper, "*X*-ray Diffraction Data on Some Radioactive Oxide Minerals (*Am. Mineral.*, **35,** 386–400, 1950).

The Council of the Walker Club wishes to acknowledge the courtesy of the Council of the Mineralogical Society of America in again making possible the publication of a Canadian number of *The American Mineralogist.*

Walter M. Tovell,
Secretary-Treasurer

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* Founded in 1938 and named in honour of the late Professor T. L. Walker (1867–1942), then Professor Emeritus of Mineralogy and Petrography in the University of Toronto and Director of the Royal Ontario Museum of Mineralogy. The by-laws of the Club were published in *Am. Mineral.*, **34,** 469, 1949.

BOOK REVIEWS

DE RE METALLICA, BY GEORGIUS AGRICOLA. Translated by Herbert Clark Hoover and Lou Henry Hoover, Dover Publications, New York. xxxii+638 pp., 1950; Price $10.00 (Originally published by the *Mining Magazine*, London, 1912).

This is not a new edition but an exact reprint of the well known Hoover translation. Plates, pagination and arrangement are identical with the 1912 edition. Only the size has been changed; the over-all size is about $\frac{8}{10}$ of that of the earlier printing (13 $\frac{3}{4}$×8 $\frac{3}{4}$″) and the plates and printed pages are about $\frac{7}{8}$ as large as in the original printing of the Hoover translation, which was very nearly identical in size with the original Latin edition.

The publishers are to be congratulated for having brought out an edition of this world famous and historically important book priced so that anyone who really wants a copy can own one. This was accomplished largely by avoiding a new type-setting job and by using not too expensive paper and binding. Although the present printing may not be as appealing to the ardent bibliophile as the earlier one, it is a very well bound volume printed on a good grade paper and will be a substantial, permanent and useful addition to the library of anyone interested in the history of technological chemistry, metallurgy, mineralogy, geology, mining or engineering. The plates have lost none of their clarity or pleasing effect by the reduction in size and the type is still large enough to be easily readable.

In addition to the complete translation of all twelve books of Agricola's original work there are three interesting and useful appendices. The first one gives a review of Agricola's principal works; a list of all his other known writings including works on other subjects, and lost or unpublished works; and bibliographic notes on contemporary and earlier works referred to in the text. Appendix B is a concise discussion of ancient authors (9 pages) and Appendix C is a discussion and list of weights and measures, with modern equivalents where these can be determined. There are 12 pages of general index, 7 pages of index to persons and authorities and a 2 page index to illustrations. Facsimiles of the original title page and of four pages of the Latin text are included.

De Re Metallica is too well known to require more than a brief reminder that it was one of the first treatises of early modern times on the above mentioned subjects, and the most complete and authoritative treatment of mining and mining engineering. It is also the best known work of its time on metallurgy, in spite of the fact that Biringuccio's very fine treatment of the subject, *De La Pirotechnia* (1540), had appeared sixteen years earlier and Agricola's discussion of some of the metallurgical and related operations was based on Biringuccio's work. *De Re Metallica* was better known and more widely used than *De La Pirotechnia* principally because the former was published in Latin, the scientific language of the day, whereas the latter appeared in Italian. Agricola's work also had many more and better illustrations than the other; it was the standard text and reference book on production of metals for almost 200 years after it was first published in 1556.

The Hoover translation is by all odds the most complete and accurate translation to be published in any modern language. The Hoovers spent five years in making the translation and expended a tremendous amount of effort in research and careful writing to insure the accuracy of the work and to preserve the original flavor insofar as this was possible. Such a task was possible only by a combination of rare linguistic ability and a thorough knowledge of mining and related sciences. The text is supplemented with copious and invaluable footnotes by the translators. These deal not only with meanings of words and references to other works by Agricola, but also give a great deal of historical and geographic background that add much to the usefulness of the book and to the reader's enjoyment of it.

This classic should be in the library of every department where any Earth science is taught and in many more general libraries that have an interest in natural history. It is to be hoped now that this work is more readily available than it has ever been before that some familiarity with its contents will be required of students rather than just a knowledge of its existence.

Earl Ingerson,
U. S. Geological Survey,
Washington, D. C.

SYMPOSIUM ON MINERAL RESOURCES OF THE SOUTHEASTERN UNITED STATES. Pages 236. University of Tennessee Press, Knoxville, Tennessee.

This being a symposium by 20 different authors on 19 types of mineral deposits, it seems advisable to give the table of contents as follows:

Geologic Investigations and Exploration in Southeastern United States	Robert A. Laurence	1
Tectonic Framework of the Southeastern States	Philip B. King	9
Problems of the Genesis of Mineral Deposits of the Southeastern States	Charles H. Behre, Jr.	26
Feldspar and Mica Deposits of Southeastern United States	John M. Parker, III	42
Geology of the Titanium-Bearing Deposits in Virginia	A. A. Pegau	49
Occurrences of Tungsten Minerals in the Southeastern States	G. H. Espenshade	56
Recent Geological Investigations in the Ducktown Mining District, Tennessee	W. W. Simmons	67
Zinc Deposits of the Southeastern States	Charles R. L. Oder and John W. Hook	72
Barite Deposits Southeast of the Appalachian Plateaus	Thomas L. Kesler	88
Kyanite and Sillimanite in the Southeastern States	A. S. Furcron	99
Talc, Soapstone, and Pyrophyllite in the Southeastern United States	Jasper L. Stuckey	112
Vermiculite of the Southeastern States	Charles E. Hunter	120
Brown Phosphate Rock in Tennessee	H. B. Burwell	128
Notes on the Land-Pebble Phosphate Deposits of Florida	J. B. Cathcart	132
Manganese Deposits of the Southeastern States	Hugh D. Miser	152
Bauxite Deposits of the Southeastern United States	Josiah Bridge	170
Economic Minerals in the Beach Sands of the Southeastern United States	Harry B. Cannon	202
Ground-water Geology in the Southeastern States	V. T. Stringfield	211
Ground water in Relation to Mining in the Southeastern States	G. D. DeBuchananne	223

The volume opens with a preliminary section by Robert A. Laurence on Geologic Investigations and Exploration in Southeastern United States. This gives a historical outline of mineral explorations in the region and emphasizes the great need for detailed geologic mapping. This is a problem of more than usual difficulty, because of the geologic complexity of the region and the exceedingly thick residual mantle covering much of it.

The section on the tectonic framework gives a very valuable outline of the geologic provinces of the region together with a map. These are discussed individually, and the complex tectonic features are presented with the help of numerous diagrams. This difficult task is done as fully as space, the complexity of the region, and an acknowledged incompleteness of information will permit.

The section on problems of genesis of mineral deposits discusses 10 classes of mineral deposits, an unusually large and varied number for any region. Under the head "Problems in Genesis," the following topics are discussed: nature, source, and movement of zinc- and lead-bearing solutions; origin, nature, and zoning of ores of the Ducktown type; sedimentary iron ores; and residual iron, manganese, and aluminum deposits.

Although the 15 papers on specific mineral deposits differ among themselves in the extent to which they analyze geologic relationships, all present an adequate discussion of mineral resources. Moreover, most of them list very full bibliographies, the paper on zinc deposits containing 80 entries. Many of the papers have excellent maps, diagrams, and plates. One map in a folder shows the very detailed geology and the complex structure of the Ducktown region.

Some of the papers are much more than statements of mineral resources and outlines of relationships. They make original contributions to our knowledge of the geology of the deposits themselves and also to a broader understanding of the geology of the region as a whole. Thus, others than those interested in the commercial possibilities of the region will have occasion to consult the volume. As an example of a paper contributing to the broader geologic relations in the region, the one on bauxite deposits by Josiah Bridge, may be mentioned.

The last two papers in the volume, the ones on Ground-Water Geology and on Ground Water in Relation to Mining, are perhaps unusual but certainly interesting and useful additions to a symposium of this type.

The volume is well printed on good paper, and is one of which the Chairman of the Symposium Committee (F. G. Snyder), those who cooperated in its organization, and the contributors may all be proud.

CLARENCE S. ROSS

(*Publication authorized by the Director, U. S. Geological Survey.*)

ANNUAL MEETING

The thirty-second annual meeting of the Mineralogical Society of America will be held in Detroit, Michigan, on November 8–10, 1951, with headquarters at the Hotel Statler.

Abstracts of papers to be presented at the annual meeting must be received by the Secretary on or before *July 16, 1951.* Abstract blanks may be obtained from the Secretary.

C. S. HURLBUT, JR., *Secretary*

A procedure is now available whereby prospectors and mine operators in the Upper Midwest may receive financial aid through the Defense Minerals Administration in their search for new ores essential to the Nation's military and civilian defense production program. Applications for assistance in Region V should be filed at the Defense Minerals Administration office at 2908 Colfax Ave. South, Minneapolis 8, Minnesota. Region V covers the States Minnesota, Wisconsin, Michigan, North Dakota, South Dakota, Nebraska, and Iowa.